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THE GENESIS OF TWIN CRYSTALS

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ABSTRACT

Interest has recently been indicated in a possible cause for the occurrence of twins. Apparently the only popular approach to twinning has been through the empirical rules of twinning enunciated by the French School. The writer's views, now some twenty years old, are herewith presented as a rational, structural approach to the cause of twinning.

Since a stable crystal represents the least energy state of a collection of atoms, it is evident that the twin boundary must correspond with a higher energy state. It is shown that this higher energy is largely associated with the higher coordination spheres of the atoms. That a twin can form at all requires that the first coordination conditions of the atoms at the boundary are satisfied. This view is purely structural and has been substantiated in twins which have been considered with regard to structure.

On the basis of genesis, twins may be divided into the categories of *growth twins*, *transformation twins*, and *gliding twins*. Growth twinning is caused by an accidental departure from equilibrium during growth, and is favored in the nuclear stage, especially under conditions of supersaturation. Transformation twinning is a consequence of high-low inversion. Glide twinning is caused by a specific type of structural shear in plastic deformation. Parting is, in part, a consequence of the high energy in the twin boundary. The act of twinning has analogies with the act of transforming from one polymorph to another: growth twinning corresponds with *reconstructive transformation;* glide twinning corresponds with *displacive transformation*. Transformation twinning is a special case of unmixing.

The oft-repeated saying that twinning represents the result of an effort on the part of the crystal to assume a greater symmetry is the exact opposite of the true state of affairs.

INTRODUCTION

In recent papers[1,2,3] some observations on the occurrence of twinning have been made and the question of what is responsible for the formation of twins has been raised. The writer's own views on this question have had a limited circulation. In view of the general interest in the subject, it seems desirable to give them wider publication.

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[1] Donnay, J. D. H., Width of albite-twinning lamellae: *Am. Mineral.*, 25, 578–586 (1940).

[2] Emmons, R. C., and Gates, R. M., Plagioclase twinning: *Bull. Geol. Soc. Am.*, 54, 287–304 (1943).

[3] Donnay, J. D. H., Plagioclase twinning: *Bull. Geol. Soc. Am.*, 54, 1645–1652 (1943).

The cause of twinning is easily approached through energy considerations. A stable crystal represents the lowest free energy state of that collection of atoms; this follows from the very notion of stability. The pattern preferably assumed by atoms assembling themselves as a crystal is thus the configuration of least free energy. Any departure from this pattern represents an increase in the energy of the collection of atoms. Since the pattern of atoms in the twin boundary represents a departure from the rest of the crystal pattern, it is obvious at the outset that the twin boundary region is a locus of higher energy than the rest of the crystal. What permits these atoms to assume this configuration of higher energy? To answer this question is to assign a cause to twin formation.

Relation of Twins to Other Kinds of Intergrowths

The energy in the twin boundary is stored as surface or interfacial energy. This kind of energy also exists in the mutual boundaries of other kinds of intergrowths. The writer has pointed out[4] that the locations of the interfaces of unmixing intergrowths is conditioned by requirements of minimum interfacial energy, and that low energy is made possible by the existence of a plane of atoms in common between the two unmixing phases. This provides a gradation in structure rather than a complete discontinuity; consequently the additional energy in the junction plane is not far above that of a plane in either crystal. This near-minimum energy requirement naturally occurs in all kinds of intergrowths and it conditions the mutual orientations of crystals in such intergrowths.[5]

Attention is now directed specifically to intergrowths of the same species of crystal. In the genesis of such intergrowths, assuming that the individuals of such pairs do not arise independently of one another, the most probable orientations are such as to provide the greatest number of structural elements in common. This structural relationship provides reduced interfacial energy. Other things being equal, the greater the number of atoms in the region of the interfacial plane which are able to participate normally in the bonding of the two structures, the less abrupt is the discontinuity, the lower is the energy, and consequently the more probable is the intergrowth. For example, a less probable intergrowth would be one in which one set of translation-equivalent atoms were normally bonded to both of the structures, but with the rest of the structure suffering a discontinuity at the boundary. The most probable intergrowths are those in which all atoms of the structure in the region of the junction bridge the junction. The bonding picture of this type of intergrowth will be discussed shortly.

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[4] Buerger, M. J., The temperature-structure-composition behavior of certain crystals: *Proc. Nat. Acad. Sci.*, **20**, 452 (1934).

[5] Royer, M. L., Recherches expérimentales sur l'épitaxie ou orientation mutuelle de cristaux d'espèces différentis: *Bull. Soc. Franc. Minéral.*, **51**, 7–159 (1928).

Gruner, John W., Structural reasons for oriented intergrowths in some minerals: *Am. Mineral.*, **14**, 227–237 (1929).

A twin is generally recognized as being a *rational*, *symmetrical intergrowth* of two individual crystals of the same species. In a very broad way, any intergrowth of two individuals of the same species is symmetrical, in the sense that one individual is a repetition of the other by some geometrical operation. When the operation conforms to the requirements for a crystallographic operation, it has conformed to the requirement for an operation which transforms a net into itself; the intergrowth is then rational. Rational intergrowths naturally have higher probabilities than non-rational intergrowths.

STRUCTURAL REQUIREMENTS FOR LOW ENERGY

These considerations have been rather general, but they are easy to translate into a structural picture. Without being specific regarding the type of bonding or the kind of structure, it can be said that the energy of an atom is a sum of a series of terms each representing the energy of that atom with respect to another atom in the configuration, the summation being carried out to all atoms in the configuration. Energy, in general, decreases very rapidly with distance; consequently the energy summation converges rapidly with distance. The nearest neighbors, therefore, are the highest contributors to the summation. Thus the first coordination sphere of an atom determines a large part of its energy, the second coordination sphere modifies this sum somewhat, and higher coordination spheres modify the energy sum to a lesser and lesser extent.

Evidently an atom can participate in a twin boundary structure and contribute a low energy to the summation provided it can merely satisfy its immediate coordination requirements. In the comparatively few cases in which the attention has been called to the relation between a structure and the transition structure inferred for the twin boundary,[6,7,8,9,10] it has been evident that the twin exists by virtue of the fact that the atoms in the structure permit more than one linking of them-

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[6] Buerger, M. J., The plastic deformation of ore minerals: *Am. Mineral.*, **13**, especially pp. 40–41 (1928).

[7] Aminoff, G., and Broomé, B., Strukturtheoretische Studien über Zwillinge, I: *Zeits. Krist.*, (A) **80**, 355–376 (1931).

[8] Taylor, W. H., Darbyshire, J. A., and Strunz, H., An x-ray investigation of the feldspars: *Zeits. Krist* (A) **87**, 490–494 (1934)

[9] Bragg, W. L., *Atomic Structure of Minerals:* Cornell University Press, Ithaca, N. Y., 52, 87, 119–121, 243–250 (1937).

[10] Zachariasen, W. H., The crystal structure of sodium formate, $NaHCO_2$: *Jour. Am. Chem. Soc.*, **62**, 1013 (1940).

selves consistent with their immediate coordination requirements, giving rise to two different configurations; one of these configurations represents the structure and its continuation, the other represents a transition to the twin configuration.

It must be inferred from this that the existence of a twin is permitted by the nature of the structure itself. *If the structure is of such a nature that, in detail, it permits a continuation of itself in alternative twin junction configuration without involving violation of the immediate coordination requirements of its atoms, the junction has low energy and the twin is energetically possible.*

This theory of the energy permissivity of twins emphasizes the characteristics of the structure itself, in contrast with the French theory of twinning,[1,3,11] which, ignoring the structure itself, merely requires certain special dimensional qualities in the identity periods of the structure. Furthermore, the French theory of twinning is strictly empirical, while the theory presented here is a rational one. Apparently the two theories are in conflict, but some relations between them will be considered further on in this paper.

TWIN FORMATION

INTRODUCTION

There appear to be at least three distinct devices which are responsible for bringing about the high energy states of twin boundaries. Corresponding with these there may be recognized several genetic types of twins: *growth twins*, *transformation twins*, and *gliding twins*. In the following sections, some aspects of the genesis of these twins are considered.

GROWTH TWINS

Geometrical aspects.—To understand how twins develop during the growth of the crystal, one must be acquainted with the mechanism of crystal growth, for twins develop as accidents of the growth process.

The general rule of crystal growth is that the newly arriving atom (or cluster of atoms) takes up a position such that the part of its normal coordination which it assumes on contact with the surface is a maximum as compared with what it would be on other parts of the surface. Thus, if the atom is the first one to arrive at a plane surface, all positions where it could continue the pattern are the same, and it can therefore assume any one of these identical positions. On the other hand, the next atom to arrive which can also coordinate with the first atom must necessarily assume a position so that it does coordinate with it; in this way, the atom improves its coordination and so loses more energy than by occupying a

[11] Friedel, Georges, *Leçons de cristallographie:* Berger-Levrault, Paris, 421–483 (**1926**).

position on the plane and away from the first atom. In a like manner, each newly arriving atom locates itself in a place touching one or more of the atoms already located on the old surface, and the process of crystal growth consists of spreading of the new layer to cover the old surface. Under near-equilibrium conditions, a second new layer does not start to form until the first new layer is completed. During the process and until a layer is completed, there is always a corner at the base of the new surface to which atoms or clusters of atoms are especially attracted because of the greatest energy loss in the form of greatest coordination offered there.

The above considerations are primarily concerned with the addition of atoms to the continuation of the normal pattern of the crystal. An atom can also fall into a position which does not continue the pattern, yet which satisfies its first coordination sphere. Should the atom fall into such a position, its energy would be an approximation to minimum energy, according to the discussion under "Structural Requirements for Low Energy." Since it does not have actually the lowest energy, however, it has a comparatively high fugacity, and it normally would lose its place for a more suitable one by a mechanism of thermal agitation and atomic bombardment.

What factors are responsible for maintaining an atom in a position of subminimum energy? A possible factor might be the simultaneous arrival at neighboring positions of a pair of atoms or more which can mutually coordinate, or, what amounts to substantially the same thing, the arrival in the twin position of a group or cluster of atoms already coordinated. The entire cluster has a smaller fugacity than a single atom, and an atom bound within the cluster by coordination also has a lower fugacity than any single atom arriving alone at a subminimum energy location. If the arrival of the cluster at the subminimum location is immediately followed by the arrival of other atoms or clusters to continue the twin pattern, a twin may persist.

We now turn to the empirical rules of twinning of the French School. Bravais[12] and Mallard[13] observed that twins appear to be possible if the lattice of a crystal has a higher dimensional symmetry or pseudo symmetry than the crystal. These additional symmetry elements are the ones which relate the individuals of the twin and constitute the elements of the twin law. Friedel[11] generalized this purely empirical rule and held that twins are possible even if a multiple cell (of small multiplicity) has a pseudo symmetry beyond the symmetry of the crystal. The occurrence of twinning, according to Friedel, is more probable the lower the "index"

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[12] See Friedel, *op. cit.*, p. 427.

[13] See Friedel, *op. cit.*, p. 434.

(which is related to the multiplicity) of the pseudo cell and the smaller the obliquity of the row in this cell to an ideally symmetrical row.

It should be possible to interpret the findings of the empirical French School on grounds of energy and with the aid of the theory of crystal

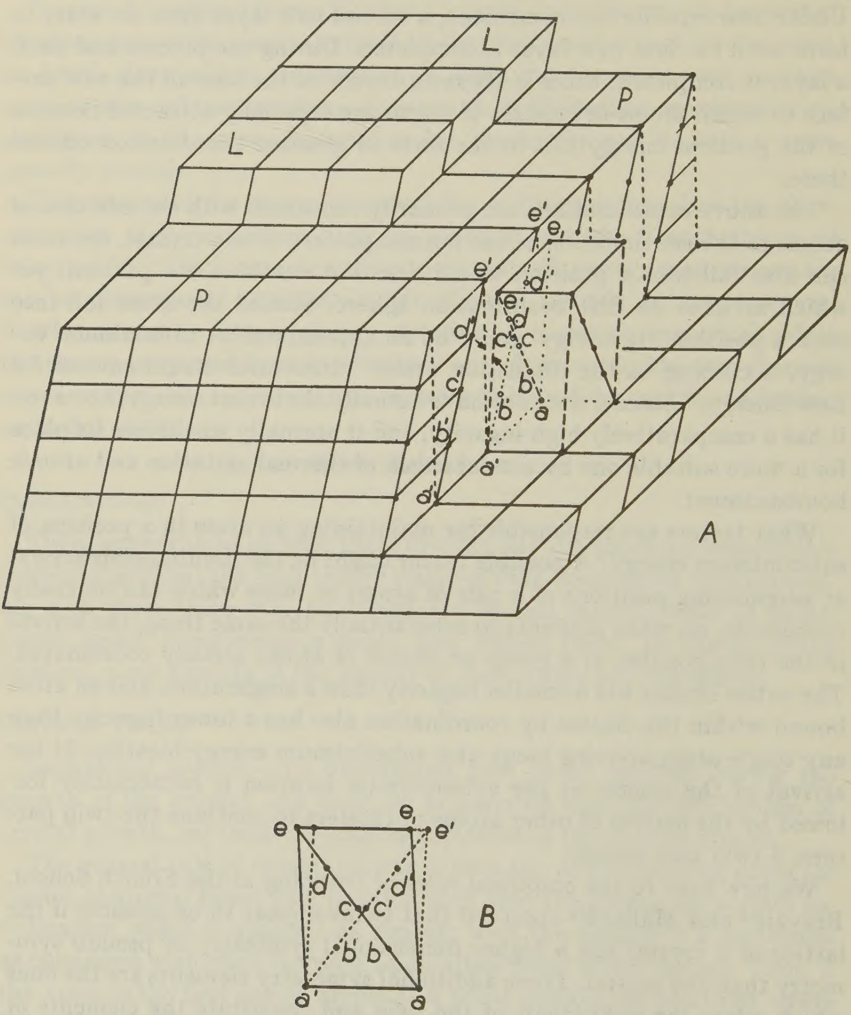

FIG. 1

growth by spreading of one layer over another. A stage in the normal growth of the crystal is represented in Fig. 1A. Here the representation of the crystal is by means of cells in accordance with the French School, not by means of structure. The crystal plane, *PP*, which is growing, is being covered by the new layer, *LL*. The cliff at the right shows how the simple cell is related to Friedel's dimensionally more symmetrical multiple cell. Now if, at any time, such a cliff is momentarily established, say, due to rapid growth, it becomes geometrically possible for several atoms (or a cluster) to add themselves to *both* surfaces of the reentrant at the foot of the cliff. The two surfaces of the reentrant provide a greater-than-normal coordination for a large cluster, especially as compared with the plane *PP* alone. Thus, should a cluster arrive in twin position, it might be able to retain this subminimum energy attachment merely because it has a multiple grip on the crystal which acts as a considerable energy barrier to removal. Once established, the pattern of the twin continues.

If this view is correct, then the interpretation of Friedel's rules must be as follows: the geometry of the lattice aids twin formation because a cluster, represented by points *abcde*, Figs. 1A and 1B, arriving at the crystal, can superpose itself in twinned orientation on the untwinned crystal, $a'b'c'd'e'$, since a small cluster is sufficiently elastic. Thus e_1 coincides with e' and c with c'. Friedel's angle of obliquity is then half the three-dimensional angle $e'ae_1$. Such strain is directly proportional to the tangent of the angle. (A continued coincidence of the two twinned parts of the crystal is unlikely since it involves the work of shearing the entire crystal through the angle of obliquity.) The significance of Friedel's low multiplicity of the multiple cell would be that as many points as possible, such as e and c, should be capable of joining the crystal and its twinned structure.

At this point it is necessary to amend the French theory.* Its rules provide, perhaps, *necessary* conditions for certain growth twins to occur, but they are definitely not sufficient conditions. The French theory, as it stands, provides only an incomplete picture of permissivity of twinning. To amend it we add the requirement of low energy junctions from twinned to untwinned *structure*, at least at the point of inception of the twin. This implies, in general, that the structure must be continuous, at least in the nearer coordination spheres of the atoms in the junction structures. It should be emphasized that this requirement is not only rational, but it has been found to hold in such twins as have been examined with regard to structure.

Environment factors favoring genesis of growth twins.—In addition to the geometrical factors permitting growth twinning, just discussed, certain external and internal environmental factors encourage the formation of structures of subminimum energy. Among these are supersaturation and minuteness of crystal.

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* There should be no hesitance in discarding or amending older crystal theories, since the crystallography before about 1915 was concerned largely with lattice geometry, especially as inferred from outer form. Present day crystallographers have the advantage of a knowledge of the details of the structures of crystals, which gives them clearer insight into all crystal phenomena. To neglect this advantage is to continue yesterday's science.

It is evident that if the rate of addition of atoms to the surface of a crystal is sufficiently high, the probability will be increased that an atom arriving at a twin position will not be relocated before it is joined by a second atom. Once a second atom joins the first, the pair has a very low energy because each member has its coordination increased by the other. Thus, once started, the twinned layer may persist and grow.

The probability of twin formation increases with the rate of addition of atoms or clusters to the crystal, and thus, within limits at least, increases with the degree of supersaturation, suggesting the genetic name *supersaturation twin*. While supersaturation may cause twinning at any time during the history of growth of the crystal, the most obvious time when the degree of supersaturation is very high is just before nuclei start to form. Once nuclei appear, the immediate regions surrounding them become cleared of a high degree of supersaturation, and a normal growth rate is established consistent with the rate of diffusion. For this reason, the condition causing supersaturation twins is most likely to arise just once—as the crystal nucleus forms—and then the conditions leading to this type of twinning do not usually occur again. Nuclei supersaturation twins are therefore characteristically simple pairs (such as Carlsbad, Baveno, and Manebach twins in feldspars). This condition is enhanced by the factor discussed in the next paragraph. Supersaturation twins *can*, however, recur. Suppose, for example, that feldspar crystals, forming in a magma, should sink to a lower region where the magma is supersaturated. The conditions there are ripe for the formation of supersaturation twins.

Special conditions obtain during the formation and early growth of a crystal nucleus which are particularly favorable to twin formation. When a crystal consists of merely two or three layers, the energy of an atom on its surface is not the same as that of the atom on the surface of an indefinitely large crystal. The energy summation consists of only a few terms rather than an infinite number and may be too small to closely approximate the converging value of the series. Conditions are thus different; they may be less unfavorable, if not positively favorable, to the formation of twins. As the crystal grows, the energy sum changes, or may even fluctuate during growth, until the crystal is large enough so that the energy sum has substantially converged. If growth now continues under equilibrium conditions, a twinned crystal has a higher energy and is subject to dissolution and reprecipitation on an untwinned crystal. If there is supersaturation, however, the chances are increased that the twinned crystals will grow to sufficiently large size that they will not readily redissolve.

TRANSFORMATION TWINS

It can be theoretically demonstrated that a pair of crystals connected by a high-low transformation possess related symmetries. The low temperature form has a symmetry which is a subgroup of the symmetry of the high temperature form. Now, in the transformation from the high form to the low form, the general structure remains the same, but some of its symmetry operations are suppressed. The low form merely forms at the expense of the high form by loss of symmetry. This can, in general, take place in such a way that the low form has more than one orientation with respect to the high form. This is indicated by the following analysis: the low form contains fewer coincidence operations than the high form. It can be transformed into itself in n ways, where n is the number of operations in its symmetry group. Since the group of the high form contains the group of the low form as a subgroup, the high form can be transformed into itself in mn ways. This means that the low form can be oriented in n indistinguishable ways in the structure while the high form can be oriented in mn indistinguishable ways in the structure. For any given orientation of the low form, there are m possible orientations of the high form. That is, there are m mutually different orientations of high and low form.

High-low transformations take place as soon as the temperature of a region has reached the transformation level, and is propagated as a wave as rapidly as heat can be transmitted. Unless the crystal is extremely small, transformation nuclei may appear in various parts of the crystal spontaneously, particularly at the edges, and the transformation spreads from these centers. With falling temperature, the chance that any center shall assume the same orientation as a neighbor is $1/m$. In general, then, a transformation from the high to the low form consists of a spontaneous formation of nuclei in m different orientations and these nuclei grow until they make contact with one another. From the mechanism of formation of these crystals, their contacts are often, though not necessarily, irregular. All individuals of the aggregate are either in parallel or different orientation. Those in different orientation are in twinned orientation with respect to one another. This follows from the fact that they could be brought into coincidence by one of the m operations of the high form which vanished in the formation of the low form.

GLIDING TWINS

It is well known that some crystals can yield to stress plastically by a process known as gliding. Two types of gliding are known, translation-gliding and twin-gliding.[14]

An untwinned crystal enjoys a state of lowest energy. If it should yield to stress by twin-gliding, then a higher energy is assumed. The energy is supplied, of course, by part of the work of deforming the crystal. In comparison, it should be noted that crystals which become twinned in the process of growth never attain a lowest energy state, while gliding twins are originally in a lowest energy state, but absorb energy in twinning and accordingly acquire a higher energy state.

The higher energy of twins is substantially localized in the close vicinity of the twin boundary. The energy is essentially a surface energy or interfacial energy since it is associated with a discontinuity or interface between two homogeneous regions. Because of the special mechanism of the gliding process, the junctions between gliding twins are characteristically plane, although they may curve slightly and delimit the twin as a thin lens, especially if bending of the crystal is involved.

If a crystal can form twins according to a certain law by growth twinning, it may or may not be able to form gliding twins according to this same law. While this will be discussed from a structural viewpoint later on, the energetics of the situation will alone be noted here. In glide twinning, each atom in the layer to start the twin is in a deep energy depression and must be caused to migrate to another depression. Energy barriers separate these depressions, otherwise they would not be depressions. If the energy barriers are comparatively low, glide twinning can be caused to occur. For example, if, for each atom, there exists a low energy path from normal position to twin position (say, a trough between depressions), then little energy is required to cause twinning shear between sheets. (For translation-gliding to take place, the energy trough must connect translation-equivalent depressions, not twin-equivalent depressions.) If, on the other hand, the depressions are surrounded by the highest energy barriers, then, to cause gliding is identical with causing the sheared sheets to separate, and thus the work of rupture (which may be cleavage) must be accomplished to cause gliding. Crystals with energy depressions so surrounded cannot be caused to glide, but rather break if sufficiently stressed. It goes without saying that the distribution of energy barriers is a function of structure.

This general situation is a great restriction to twinning by gliding.

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[14] Buerger, M. J., Translation-gliding in crystals: *Am. Mineral.*, **15,** especially pages 46–47 (1930).

Growth twinning is unrestricted by such potential barriers since each atom or cluster merely adds itself to the structure from a position in space which has a higher energy than either the normal location or the twin location. Thus many twin laws which are ordinarily possible for growth twins are impossible for gliding twins.

When a crystal yields by twin-gliding, it does so in a peculiar sequence, and the process ordinarily leaves a characteristic record of its occurrence. If a stress having a shearing component in the glide plane is applied to the crystal in sufficient magnitude to cause overriding of the barriers, all parallel planes in the crystal are subject to the stress, yet gliding does not occur on all planes at once. What happens is that some fortuitous circumstance, possibly a flaw, defect, or even heat motion, reduces resistance locally. The crystal yields in this region and a twin lamella of minimum size forms delimited by two twin boundaries. In the neighborhood of these boundaries the energy is high, and consequently these regions are relatively yielding. Thus, with continued stress, gliding takes place in the next sheet to each twin boundary, and the twin lamella grows in width. Ordinarily, the lamella does not grow indefinitely, but is either blocked by a flaw, or fortuitous circumstances raise the resistance to shear in the original twin boundaries until resistance in some untwinned region is lower. When this occurs, further yielding by the crystal occurs by yield in a new place and there results the development of a new lamella. A crystal which has yielded plastically by twin-gliding is accordingly characterized by containing numerous parallel twin lamellae or bands. This repeated twinning is necessarily alternating twinning, and thus is polysynthetic twinning. The distribution of lamellae can be anything from regular to irregular, depending on the distribution and character of the "fortuitous circumstances" mentioned above. The widths of the bands depend on the extent of the deformation and distribution of "fortuitous circumstances."

MISCELLANEOUS FEATURES OF TWINNING

Parting and Replacement.—It has already been pointed out that a twin boundary is the locus of high energy. The work required to disrupt the crystal is partly represented by this increase in energy of the crystal at the boundary. If the boundary is plane, it is the weakest individual plane of all parallel planes, and if tension is applied to the crystal, the crystal breaks at the twin boundary (excluding cleavage). This constitutes parting. Thus twin boundaries are parting planes. (Other causes of "parting" are also recognized.[15]) The high energy aspect of the twinning plane also localizes chemical action, so that chemical alteration and replacement tend to occur preferentially at twin boundaries. Such alteration, of course further weakens the resistance of the twin boundary to stresses and enhances the ease of parting.

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[15] Greig, J. W., Merwin, H. E., and Posnjak, E., Separation planes in magnetite: *Am. Mineral.*, **21**, 504–510 (1936).

Relation of Twinning to Polymorphism.—Anyone who has been concerned with the mechanism of twin-gliding[6] has noticed that the structure in the twin boundary may be a polymorphic modification of the original structure. Aminoff and Broome[7] have discussed the case of twinned sphalerite, which has the wurtzite arrangement in the twin boundary. Other examples could easily be cited. Indeed it is true in general that the structure in a twin boundary is at least a possible polymorphous alternative structure. This is because the atoms of most (though not all) polymorphous pairs have identical first ocordination spheres (some polymorphous pairs have different first coordination spheres) but different higher coordination spheres. This is exactly the situation in the twin boundary. In this sense, twin boundaries are cases of two-dimensional polymorphism. Thus the high energy of a twin boundary is related structurally to the high energy of an unstable polymorphous form.

This suggests the following hypothesis: the possible growth twin laws of a crystal truly unstable under the conditions of its formation can include only such laws as have boundary structures of less stable polymorphic forms, and cannot include twin laws which have boundary structures of the stable modification. (Otherwise, the structure of the stable modification once started, would perpetuate the stable modification and the twin would cease to exist since the intergrowth would be one between stable and unstable modifications.) Should such a twin law be observed in an unstable crystal, the crystal is not a growth twin, or else the modification was not actually unstable under the conditions of formations. The possibilities of twinning in unstable crystals are thus restricted, and, in general, an unstable modification should display fewer twin laws than a stable modification.

Relation of Twin Formation to Polymorphic Transformation.—Not only is twinning related to polymorphism, but a change from untwinned to twinned condition (as in glide twinning) is related to polymorphic transformation. From a structural point of view, two important kinds of polymorphic transformation can be recognized:

(1) Transformations involving only a slight displacement of parts of the structure, of the order of heat motion. This kind of transformation may be termed a *displacive transformation*. It is of the high-low type and is called forth instantly by transgression of the critical temperature (example: low-quartz to high-quartz).

(2) Transformations requiring a bit-by-bit tearing down of one structure and a rebuilding of the bits to form a differently linked structure. This may be called a *reconstructive transformation.* It is of the sluggish variety because it requires a distillation of one structure into the other (example: quartz to cristobalite). Such transformations cannot be effected by simple displacement.

There are also displacive and reconstructive varieties of twinning. In the *displacive* variety, the crystal and its twin boundary patterns may be made to transform into one another by simple displacement. Accordingly such twins can be formed by a high-low transformation or by twin-gliding. The possibility of a displacement is the structural equivalent of a low energy trough connecting energy depressions mentioned above.

Other twinning is of the *reconstructive* variety, that is, the crystal can be transformed into its twin boundary only by a complete disintegration into bits and then reconstruction of its structure. Obviously, such twinning cannot result from high-low transformation or from the simple shear displacement of twin-gliding. Such twins can only be formed by the original precipitation of the twinned structure, and consequently *reconstructive twins are limited to growth twins.* This corresponds closely to the requirements for a reconstructive transformation.

It might be pointed out that the albite twinning of plagioclase is of the displacive type, since the triclinic character of this crystal is due to a collapse of the monoclinic orthoclase structure such that oxygens coordinate with the small sodium atom. No relinking of the structure is required in passing from untwinned to twinned structure. It is thus possible that albite twins can arise by either transformation twinning or glide twinning, as well as by growth twinning. Guided by this reasoning, the writer and his students have experimentally produced polysynthetic twinning in albite by glide twinning.

Relation of Transformation Twins to Unmixing.—In another place the writer has explained the formation of segregate phase crystals in unmixing.[16] These are called forth in response to a requirement for ordering in the structure as the temperature falls. Transformation twins are also called forth by falling temperature, and often, if not always, this is equivalent to a requirement for some kind of ordering. Transformation twining is merely a degenerate case of segregate phase formation in which both original disordered and final ordered phases have identical chemical compositions.

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[16] Buerger, M. J., The temperature-structure-composition behavior of certain crystals: *Proc. Nat. Acad. Sci.*, **20**, 444–453 (1934).

Remarks about Common Statements of the Cause of Twinning

One commonly finds statements equivalent to the following: "Twinning may be regarded as an unsuccessful attempt to establish a symmetry higher than that to which the simple crystal belongs." This kind of statement misses the meaning of symmetry and misses the significance of twinning.

Symmetry, in essence, is a relation equivalent to the repetition of some geometrical feature. Any two similar things form a symmetrical pair, in a broad sense, since one is the repetition of the other. Crystallographic symmetry is merely a restriction of this notion to cases consistent with lattice translations. There is no virtue in symmetry such that atoms, coming together to form a crystal, should strive to attain it. There is no potential favoring it. Indeed, in the case of twins, the potential favors its absence.

The writer is indebted to Dr. George Tunnell and Dr. J. D. H. Donnay for kindly reading this paper in manuscript and for offering constructive criticism of certain points raised in it. This should not be construed as necessarily committing them to the writer's views.

CATTIERITE AND VAESITE: NEW Co-Ni MINERALS FROM THE BELGIAN CONGO

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ABSTRACT

The group which includes CoS_2, NiS_2, and FeS_2 forms an isostructural series following the pyrite lattice. Heretofore, the formation of the artificial end members of the series has been established, but approximations to CoS_2 and NiS_2 have not been found in nature. At the Shinkolobwe mine in the Belgian Congo, CoS_2 having a pyrite structure is now known. To this mineral is assigned the name *cattierite*. Material approaching NiS_2 is composition and having a pyrite type of lattice has been found in the Kasompi mine of the Belgian Congo. This mineral is tentatively called *vaesite*. The lattice constants for the natural end members of the series in angstroms are pyrite $5.40667 \pm .00007$, vaesite $5.66787 \pm .00008$, and cattierite $5.52346 \pm .00048$. The minerals are named after important contributors to the mineral development of the Belgian Congo. The four members of the pyrite series would be vaesite, cattierite, bravoite, and pyrite.

INTRODUCTION

In September 1943 Mr. Johannes Vaes of the Union Minière du Haut Katanga submitted two metallic sulfide minerals upon which he had previously made independent mineralogical studies including chemical analyses and an examination of polished surfaces. One sample was a nickel sulfide with an analysis corresponding to the formula NiS_2; the other a cobalt sulfide corresponding to CoS_2. The nickel di-sulfide had been obtained in a core during the progress of diamond drilling at the Kasompi mine about 70 kilometers west-southwest of Kambove in Haut Katanga of the Belgian Congo. The cobalt sulfide was from the Shinkolobwe mine near Jadotville, also in Haut Katanga. Preliminary chemical and microscopic investigation had led Mr. Vaes to the interpretation that the two minerals belonged to the pyrite group and there was good reason to believe represented new species. In the interval which has elapsed since the specimens were first submitted, the two minerals have been studied in the laboratory at Columbia University. The following account is intended to set forth the results of this examination.

The identity of the two sulfides has been confirmed and it seems appropriate to suggest that they represent species worthy of special names. It seems proper that the cobaltian sulfide receive the name *cattierite* after Mr. Felicien Cattier, Chairman of the Board, Union Minière du Haut Katanga, who has had a life-long connection with colonial affairs of the Belgian Congo and whose activities have contributed so largely to the stimulation of wartime metal production. He has also taken a great interest in scientific research and the welfare of Belgian universities. It is proposed that the nickelian sulfide receive the name *vaesite* after Mr. Johannes Vaes, mineralogist for the Union Minière du Haut Katanga of the Belgian Congo, who has shown such a keen interest in the study Katanga minerals.

RS_2 COMPOUNDS

Identification of the two minerals in question depends to a considerable degree upon comparison with material previously described. This includes both minerals and artificial compounds of the RS_2 type which contain cobalt, nickel, and iron.

ARTIFICIAL CO-NI-FE DISULFIDES

It is reported that CoS_2 was made by von Setterberg (1826), although the melting and dissociation points of the synthetic product were not determined. Von Fellenberg (1840) is credited with having made NiS_2 in a melt of $NiCO_3$ and K_2CO_3. Both syntheses were completed before the days of adequate metallographic or x-ray studies.

de Jong and Willems (1927) report the successful synthesis of CoS_2 and NiS_2. Powdered NiS or CoS under atmospheric pressure were heated 30–40 hours with a mixture of molten sulfur at 170°, followed by heating at 150°C. for 24 hours. The nickel sulfide formed contained $Ni = 45.6\%$ and $S = 54.4\%$, as compared with a theoretical $Ni = 47.8\%$ and $S = 52.2\%$. X-ray diffraction measurements by de Jong and Willems (1927) yielded determinations of the dimensions of the unit cell as follows: $NiS_2 = 5.74$ and $CoS_2 = 5.64$ Å. The specific gravities were: $NiS_2 = 4.31$ and $CoS_2 = 4.55$. Both synthetic products were reported to yield an x-ray diffraction pattern of the pyrite type.

In a recent paper Michener and Yates (1944) have called attention to a series of synthetic fusion products covering the range from FeS_2 to NiS_2. As reported, polished surfaces and x-ray diffraction patterns indicate that the intermediate products synthesized were mechanical mixtures, although the end-members were homogeneous with the pyrite type of structure. This apparently represents a set of synthetic products differing in character from homogeneous minerals of intermediate composition which show lattice variation as recorded in the work of Bannister (1940).

MINERAL CO-NI-FE DISULFIDES

Although it would appear from the work of de Jong and Willems (1927) that artificial CoS_2 with the structure of pyrite may be prepared, the existence of this material in nature has not heretofore been confirmed. The same appears to be true for naturally occurring NiS_2. On the other hand, compounds of the pyrite type in two instances containing slightly more nickel than iron, but usually containing less nickel than iron, are well established. Pyrite may occur free from nickel or with small amounts distributed through the unit cell. The nickel-bearing mineral may be known as nickelian pyrite where the amount is small, or bravoite where it is considerable. The point at which nickelian pyrite ends and bravoite begins must of necessity rest on an arbitrary decision.

Hillebrand (1907) in studying small isometric crystals associated with the vanadium ores of Minasragra, Peru, discovered a pyrite-like material which contained about 18 per cent nickel. He made a complete analysis and suggested the name bravoite. The formula given was $(Fe, Ni)S_2$ with the ratio of nickel to iron about 3 to 5. Later Hewitt (1909) in a general description of the ores showed a photomicrograph of a polished surface in which the roughly square outlines of bravoite crystals appeared to stand out in moderate to strong relief from a soft ground mass of patronite. The crystals pictured would measure up to one and one-half millimeters in cross section.

Villamanite from Villamanin station near Carmenes province of Leon, Spain, has been described by Schoeller and Powell (1920) as a member of the pyrite group. The mineral has been given the formula (Cu, Ni, Co, Fe)S, Se. It is a black mineral, granular and crystalline, which occurs in dolomite. Isometric crystals have been observed. In the absence of *x*-ray and microscopic data, the role of the mineral is uncertain.

Buddington (1924) identified a nickel-iron sulfide from the vicinity of Chitina, in the Copper River region, Alaska, which corresponded to the bravoite of Hillebrand. The Chitina material contained substantially more nickel with the iron to nickel ratio being roughly of the order of 4 to 5. Buddington also compared the properties of the two on polished surfaces as follows:

	Bravoite, Chitina, Alaska	*Bravoite, Minasragra, Peru*
Color:	dull gray	pallid gray (violet hue)
Cleavage:	3 directions	3 directions
Hardness:	4½	5 (a little less)
HNO_3:	effervesces	effervesces (turns blue gray)
HCl:	turns yellow (rubs clean)	negative
KCN, $FeCl_3$, $HgCl_2$:	negative	negative
KOH:	negative	slight reaction
Sp. Gr.:	4.31	4.33 (?)

Henglein (1914) described a mineral belonging to the pyrite group with the formula $(Co,Ni,Fe)S_2$. The mineral was found at Musen in Westphalia, Germany, and occurred in small steel gray pyritohedral crystals. Through crystal measurement the forms {023}, {001}, and {111} were identified.

Kalb and Meyer (1926) mounted crushed ore from Mechernich in a cementing medium, polished the briquettes and studied them with a microscope. Small isometric crystals with cross sections indicating a pyritohedral habit were observed. Goniometric measurements of isolated crystals showed the forms {750} and {320}. The crystals exhibited a distinct zonal banding on polished surfaces. Yellow nickel-poor bands were nickelian pyrite, while steel gray bands containing 3.28 per cent cobalt, 24.73 per cent nickel and 17.00 per cent iron were classed as bravoite. Later de Jong and Willems (1927) examined the bravoite from Mechernich which contained 24.73 per cent nickel and 3.28 per cent cobalt by means of *x*-rays. The cell edge was determined as 5.57 A.U., and the pattern was of the pyrite type.

Scholtz (1936) on the basis of microscopic study reported bravoite from the magmatic nickel-bearing ore deposits of East Griqualand and Pondoland in Africa. The mineral was observed on polished surfaces where it replaced pentlandite and vallerite. Bannister (1940) described bravoite from the Mill Close mine, Derbyshire. The mineral contained a little less nickel in proportion to iron than the Peruvian bravoite. The specific gravity was also higher, 4.82. From *x*-ray studies Bannister concluded that the mineral had the pyrite structure wth the unit-cell-edge equal to 5.49 A.U. Another bravoite in the same mine was said to contain 28 per cent nickel with a cell edge of 5.47 A.U.

While pyrite is one of the oldest of our common minerals and was recognized in the early Greek literature, the discovery of nickel-bearing pyrite is more recent. Cobalt-nickel pyrite was reported from Canada by Hunt (1866) and was later listed by Vernadsky (1914). Thomson and Allen (1939) in connection with studies of the nickelian pyrite from the Denison mine of the Sudbury district in Canada called attention to the existence of several localities in which pyrite contains a small amount of nickel. A sample from the Denison mine yielded 6.50 per cent nickel; from the Emmens Metal Company 3.5; from the Shepherd mine 5.40; and from the Murray mine 4.34. Material described by Emmens (1892) may have been of the same type. Later Peacock and Smith (1941) made *x*-ray precision measurements of common pyrite and nickelian pyrite in order to study the variation in structure with composition. They furnished a correlation of cube-edge measurements as determined by *x*-ray measurements.

X-RAY STUDY

X-ray diffraction patterns of the Debeye type were taken of both the cobaltian and nickelian sulfides and associated metallic minerals. Measurements of pyrite free from nickel and cobalt, and the nickel and cobalt sulfides of the pyrite type are shown in Table 2. Lattice constants based on the measurements of the powder diffraction patterns are included in the data shown in Table 1. The associated sulfides, aside from pyrite and chalcopyrite, are shown by x-ray diffraction patterns to belong to the linnaeite-polydymite group with the formula R_3S_4 and the spinel type of lattice. In the absence of chemical analyses of the R_3S_4 minerals no attempt is made at placement in the group.

In addition to photographs with the Debye type cameras recorded in Table 2, precision photographs were taken in a symmetrical focusing camera of the type described by Jette and Foote (1935). The latter have been computed as set forth in a separate paper on the lattice constants written in cooperation with Mr. Ralph Holmes and Mrs. Margaret Knox.

TABLE 1. SUMMARY OF LATTICE CONSTANTS

Series (Co, Ni, Fe)S_2

Mineral	Locality	a_0	Per Cent Ni	Per Cent Co	Reference
Pyrite	Leadville, Colo.	5.40667 (1)	—	—	
Pyrite	Rio Morina, Elba	5.406	—	—	Peacock and Smith (1941)
Pyrite	Leadville, Colo.	5.408	—	—	Peacock and Smith (1941).
Nickelian pyrite	Denison mine	5.419	6.50	—	Peacock and Smith (1941).
Bravoite	Mill Close Mine	5.49	16.69	—	Bannister (1940).
Bravoite	Mechernich, Germany	5.57	24.73	3.28	de Jong and Willems (1927).
Vaesite	Kasompi, Katanga	5.66787 (2)	41.24	3.41	
Artificial NiS_2	—	5.74	45.6	—	de Jong and Willems (1927).
Cattierite	Shinkolobwe, Katanga	5.52346 (3)	1.53	40.6	
Artificial CoS_2	—	5.64	—	—	de Jong and Willems (1927).

(1) ±.00007 (2) ±.00008 (3) ±.00048

Table 2. *X*-Ray Diffraction Measurements of Two Nickelian and Cobaltian Minerals Compared with Pyrite

$(h^2+k^2+l^2)$	Miller Indices	I*	Pyrite (FeS_2) *d*	Cattierite (CoS_2) *d*	Vaesite (NiS_2) *d*
3	(111)	84	3.080	—	3.238
4	(200)	100	2.673	2.750	2.809
5	(210)	60	2.394	2.463	2.515
6	(211)	48	2.186	2.249	2.298
8	(220)	34	1.896	1.950	1.992
9	(221)	40	—	—	1.902
11	(311)	55	1.618	1.663	1.702
12	(222)	17	1.550	1.592	1.629
13	(230)	17	1.490	1.529	1.566
14	(231)	22	1.436	1.474	1.509
16	(400)	—	—	—	—
19	(331)	14	1.238	1.267	1.297
20	(420)	6	1.204	1.235	1.265
21	(421)	5	1.175	1.205	1.234
22	(332)	3	1.148	1.178	1.201
24	(242)	17	1.100	1.128	1.155
26	(150)	—	—	—	—
27	(333)	55	1.038	1.063	1.080
29	(520)	—	1.002	1.026	1.041
30	(521)	—	.986	1.009	1.035
32	(440)	—	—	.977	1.002

* Intensities on the basis of photo-cell measurements.

Polished Surfaces

The cobaltian mineral from Shinkolobwe forms a granular intergrowth with another sulfide, but it may be distinguished by a cubic cleavage and pinkish color. The associated sulfide lacks the cleavage and yields *x*-ray diffraction patterns of the spinel lattice type. According to qualitative tests it is a sulfide of Co, Ni, and Fe. It is believed to be a member of the linnaeite-polydymite series of the R_3S_4 type. The mineral is a pale corinthian pink of the color shown in the color dictionary of Maerz and Paul (Plate 3, 1B, 1930). The two sulfides occur in the dolomitic gangue.

The nickelian mineral from Kasompi occurs in crystals disseminated through dolomite. These are both octahedral and cubic in habit. The cleavage is cubic and on polished surfaces the color is gray. Specimens develop a coarse cleavage pattern. Associated with the nickelian mineral is a member of the linnaeite-polydymite series. The mineral yields qualitative tests for Co, Ni, and Fe, and is a metallic sulfide. According to x-ray diffraction patterns it has the spinel type of structure. On polished surfaces it is a pale corinthian pink but does not show cleavage. Presumably it represents a mineral of the R_3S_4 type. Small crystals of pyrite are also scattered through the ore. These are euhedral with a pyritodehral habit and may be isolated from the associated sulfides. The gangue material is chiefly dolomite. It is easily disintegrated with acid and small crystals of the nickelian sulfide may be isolated from the decomposed carbonate.

Photomicrographs of polished surfaces of the two minerals of the pyrite series are shown in Figs. 1 and 2. The cleavage of each is clearly shown in the area photographed.

Polished surfaces of both the mineral from Kasompi and the mineral from Shinkolobwe were tested electrochemically by the print method in order to confirm the distribution of cobalt, nickel, and iron throughout the area of the polished surface.

In the case of the Shinkolobwe material the minerals of the polished surface are too intergrown to make more than general application of the print method. Strong qualitative tests for cobalt and iron are furnished by most of the metallic area of the polished surface. Nickel is also broadly distributed but the test secured was not as strong.

The tests on the Kasompi material were less satisfactory due to the irregular distribution of the metallic constituent throughout the gangue. Crystals of the metallic minerals were found to be isolated in a number of instances and hence not in a position to complete the circuit. Also the dolomitic gangue was strongly reactive to the iron test while the Kasompi nickel mineral hardly reacted at all. The R_3S_4 mineral associated with the Kasompi nickel mineral reacted for iron.

On the whole the print tests merely confirm the other observations to the effect that qualitatively cobalt, nickel, and iron are probably present in each of the gray metallic minerals of the polished surfaces. The texture is not sufficiently coarse and well defined to employ the method to best advantage.

NOMENCLATURE

The minerals under discussion furnish an interesting problem in nomenclature. The (RS_2) sulfides of pyritohedral symmetry with varying amounts of nickel, cobalt and iron have been described under the names pyrite, nickelian pyrite, and bravoite. The (R_3S_4) sulfides of the same elements with a lattice of the spinel type have been described under the names linnaeite, siegenite, polydymite, and violarite.

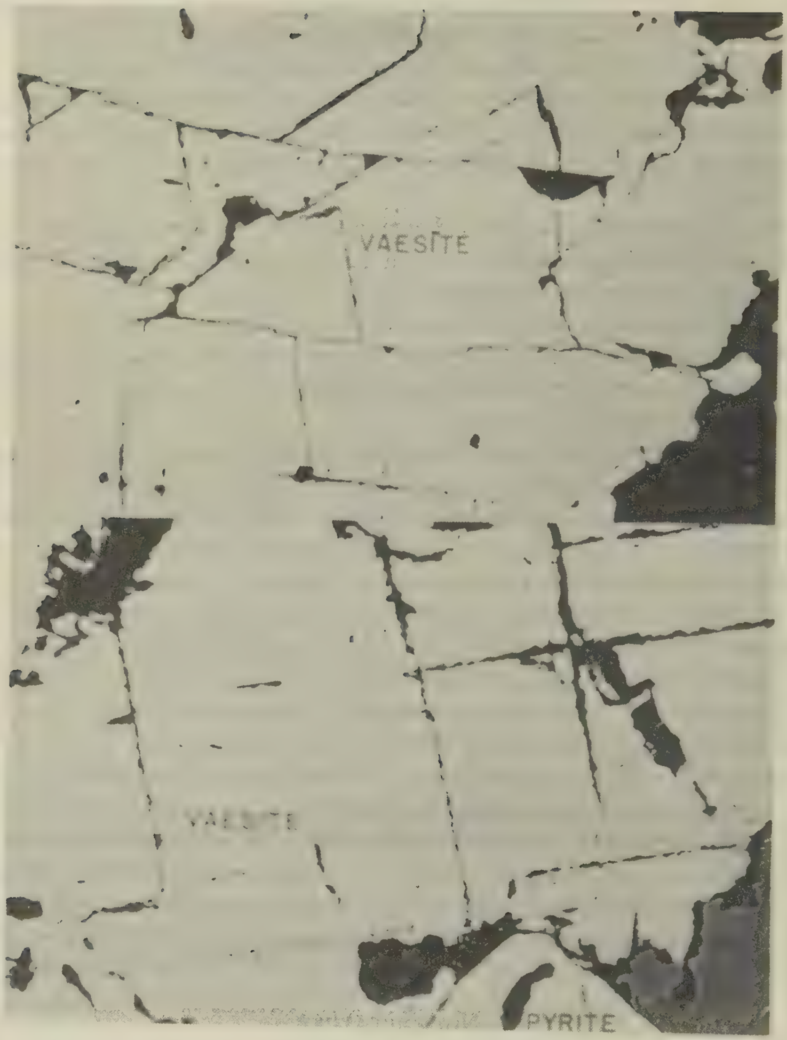

FIG. 1. Photomicrographs of a polished surface of vaesite NiS_2 from Kasompi showing well developed cubic cleavage resulting in both triangular and rectangular patterns. (Magnification ×180.)

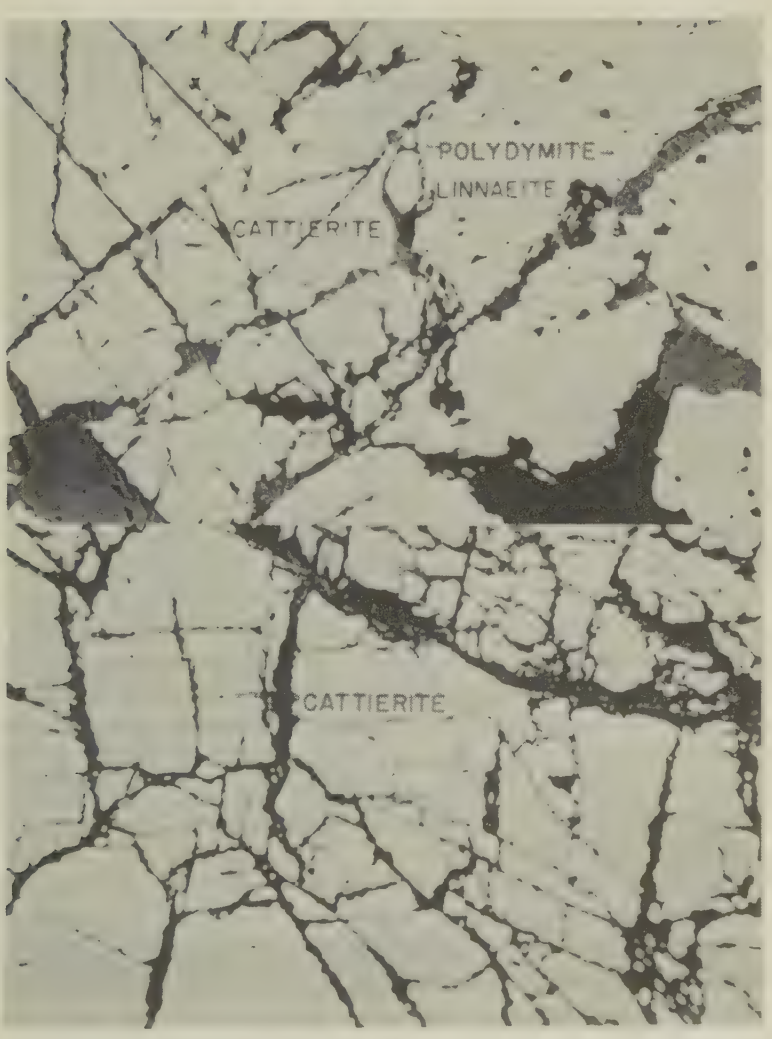

Fig. 2. Photomicrographs of a polished surface of cattierite CoS_2 from Shinkolobwe showing irregularly developed cubic cleavage. An area free from cleavage in the upper view represents a member of the linnaeite-polydymite series. (Magnification ×180.)

Where minerals belong to isomorphous groups, the distinction between species of like group structure is of necessity arbitrary. Palache, Berman, and Frondel (1944) in their review of the series NiS_2-FeS_2 propose to apply the name *bravoite* only to members which contain more nickel than iron. Specimens containing less nickel than iron would be called nickelian pyrite. Under this system of nomenclature the original bravoite of Hillebrand (1907), as well as samples high in nickel described by Henglein (1914), referred to by Doelter and Leitmeier (1926) and mentioned by Bannister (1940), would no longer qualify as bravoite but would be known as nickelian pyrite. It is assumed that a parallel system of nomenclature would apply in the case of cobalt, and any member of the group with less cobalt than iron would be cobaltian pyrite. Presumably a member of the series with an excess of cobalt would also be called bravoite, although on this point the system to be applied is indefinite.

The usage suggested is somewhat at variance with the literature. In the analyses of Table 1, according to accepted usage, bravoite has been recognized in five well-established localities. The work of Hillebrand, de Jong and Willems, Buddington and Bannister has established bravoite as a mineral with definite properties, isostructural with pyrite but containing substantial amounts of nickel. At the same time through the work of Thomson and Allen, and Peacock and Smith nickelian pyrite with smaller amounts of nickel has been recognized. The line between the two has not been definitely established, but the usage of Bannister, a recent contributor, would class material on the order of 36.3 Ni to 63.7 Fe as bravoite and no objection is expressed to considering material with a lower nickel content as bravoite. Even an excess above 20 Ni to 80 Fe would have a significance which would warrant more than adjectival description, particularly when accompanied by a definite change in lattice constant and physical properties.

It is believed this report shows that NiS_2 and CoS_2 are more than artificial end-members. Minerals with compositions close to the theoretical values are now known. Under these circumstances a triangular relationship of the type illustrated in Fig. 3 seems more appropriate as a guide to the nomenclature.

CoS_2 and NiS_2 each occupy a vertex of the diagram with FeS_2 occupying the third. Chemically the mineral from Shinkolobwe fits nicely into the cobalt corner. At the nickel corner the mineral from Kasompi corresponds as closely as one would expect a mineral to agree with the theoretical NiS_2. Thus there is good reason to seriously consider the claim of Mr. Vaes that the two minerals might properly be considered valid species.

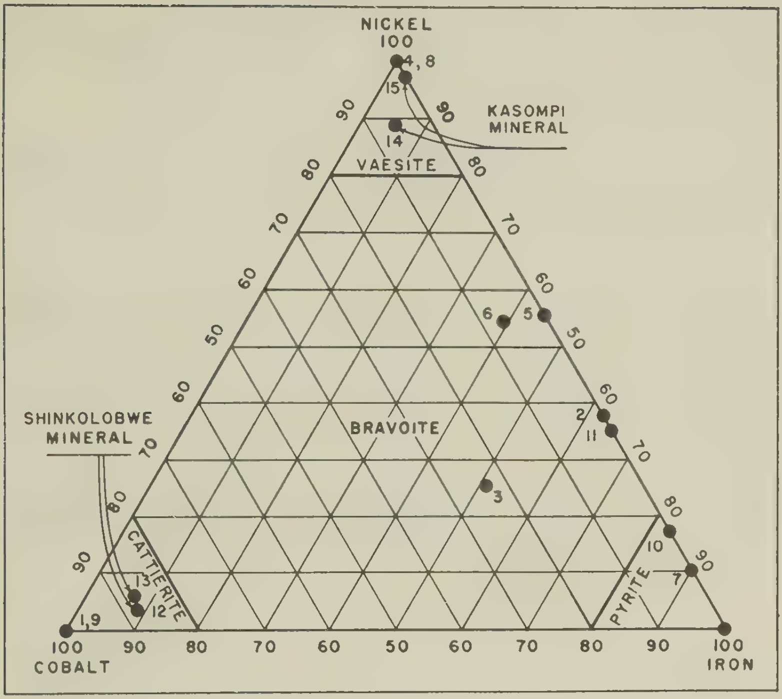

FIG. 3. A triangular diagram indicating the relative proportions of nickel, cobalt, and iron in various members of the pyrite group.

1. Artificial CoS_2, von Setterberg (1826).
2. Bravoite, Minasragra, Peru, Hillebrand)1907).
3. Bravoite, Musen, Henglein (1914).
4. Artificial NiS_2, Fellenberg (1840).
5. Bravoite, Chitina, Alaska, Buddington (1924).
6. Bravoite, Mechernich, Kalb and Meyer (1926).
7. Nickelian pyrite, Mechernich, Kalb and Meyer (1926).
8. Artificial NiS_2, de Jong and Willems (1927).
9. Artificial CoS_2, de Jong and Willems (1927).
10. Nickelian pyrite, Denison Mine, Thomson and Allen (1939).
11. Bravoite, Mill Close Mine, Bannister (1940).
12. Cattierite, Shinkolobwe Mine, Ledoux and Co. (1944).
13. Cattierite, Shinkolobwe Mine, Vaes (1943).
14. Vaesite, Kasompi Mine, Ledoux and Co. (1944).
15. Vaesite, Kasompi Mine, Vaes (1943).

If one should arbitrarily truncate the species of the triangular diagram at a line corresponding to 80%, the end members would be clearly delineated. In the center of the diagram is a comparatively large area which seemingly contains too few examples as yet to justify subdivision. Since bravoite is well established and has been confirmed from several sources, it is suggested that this name be retained for the compositions represented by the entire central area of the triangle. Thus the RS_2 triangular domain would be divided into the three vertex areas in which occur pyrite, cattierite, and vaesite, while the entire interior would be bravoite.

If the same treatment is applied to the R_3S_4) group, which has the spinel structure, representatives among minerals may be found for the Ni and Co vertices. Here the Ni-vertex would appear to warrant the name polydymite and the Co-vertex the name linnaeite. Artificial Co_3S_4 with spinel structure has been made, but artificial Ni_3S_4 is yet to be confirmed. The Fe-vertex does not appear to be represented among minerals and artificial Fe_3S_4, although claimed, has not been confirmed. The large central area would contain the R-proportions for minerals previously described as siegenite, violarite, and linnaeite. This diagram is much less definite than the one for RS_2.

Table 3. Chemical Analyses of Nickelian Pyrite and Bravoite

	Nickelian Pyrite		Bravoite				
	Denison mine (10)	Mechernich (yellow) (7)	Mill Close mine (11)	Minasragra, Peru (recast) (2)	Musen, Germany (3)	Chitina, Alaska (5)	Mechernich (steel gray) (6)
Co	—	—	—	tr	10.6	—	3.28
Ni	6.50	4.40	16.69	18.23	11.7	24.81	24.73
Fe	34.54	38.02	29.30	29.46	22.8	20.68	17.08
Cu	—	2.80	—	—	—	—	0.47
S	51.39	50.77	53.40	52.31	53.9	54.51	51.15
Insol.	3.80	2.50	—	—	0.7	—	0.40
	96.23	98.49	99.39	100.00	99.7	100.00	97.11
Ni:Fe	15.8:84.2	10.5:89.5	36.3:63.7	38.2:61.8	—	54.6:45.4	—
Co:Ni:Fe	—	—	—	—	23.5:26.0:50.5	—	7.3:54.9:37.8

Chemical Composition

Chemical analyses of cattierite and vaesite are shown in Table 4. One analysis of each mineral is substantially complete. Both confirm the cobaltian or nickelian nature of the two sulphides. The impurities for each are quartz, dolomite, calcite, mica, and chalcopyrite. The sulfides analyzed were checked by x-ray diffraction patterns which yielded the pyrite type of structure. Additional lines due to other metallic minerals were not observed. The material analyzed by Ledoux and Co. was hand picked beneath a binocular microscope, grains showing an absence of cleavage being eliminated. Since both minerals show good cleavage this property was helpful in each case in making a separation. The material analyzed by Mr. Vaes was selected on the basis of examination on polished surfaces.

TABLE 4. CHEMICAL ANALYSES OF THE COBALTIAN AND NICKELIAN SULFIDES FROM KATANGA

	Cattierite Shinkolobwe mine		Vaesite Kasompi mine	
	Ledoux & Co. (1944)	Vaes (1943)	Ledoux & Co. (1944)	Vaes (1943)
	(12)	(13)	(14)	(15)
Co	31.60	41.40	2.82	—
Ni	1.19	3.20	32.93	44.58
Fe	3.40	3.27	1.85	0.95
Cu	.34	0.59	0.12	—
S	42.25	51.32	42.61	51.22
SiO_2	.12	0.08	0.48	—
Al_2O_3	0.52	—	0.28	—
$CaCO_3$	7.98	—	2.14	—
$MgCO_3$	7.63	—	13.89	—
	95.03	99.86	99.12	96.75

TABLE 5. RECAST ANALYSES; CATTIERITE AND VAESITE

	Cattierite		Vaesite	
	(12)	(13)	(14)	(15)
Co	40.60	42.20	3.41	—
Ni	1.53	3.25	41.24	46.04
Fe	3.97	2.80	2.20	.96
S	53.90	51.75	53.15	53.00
	100.00	100.00	100.00	100.00
Computed Sp. Gr.	4.80		4.45	

A recast of each analysis appears in Table 5. The nickelian mineral vaesite yields a comparative ratio of Co:Ni:Fe amounting to approximately 7.2:88.0:4.8 according to one analysis and —:98.0:2.0 according to the other. While it is not pure NiS_2 according to either analysis, it is close to the nickel apex of the triangle. The cobaltian mineral yields ratios of Co:Ni:Fe of approximately 88.1:3.8:8.6 and 87.5:6.7:5.8, according to the two analyses. Thus it would lie close to the cobalt apex of the triangle.

ACKNOWLEDGMENTS

It is a pleasure to acknowledge the assistance of Mr. Ralph J. Holmes and Mrs. Margaret S. Knox in connection with the assembly of *x*-ray data used in this report. The cooperation of Ledoux & Co. in supplying analyses of selected material is also greatly appreciated. The courtesy of the officials of the Union Minière in supplying material for study at a time when communication is difficult has made possible the completion of work, which would otherwise have been greatly delayed.

CONCLUSION

On the basis of microscopic examination, chemical analysis, and *x*-ray study the two sulfides examined have been identified as lying close to NiS_2 and CoS_2 in the pyrite group. If the principle of nomenclature is adopted that the compositions corresponding to the apices of the triangular diagram are worthy of designation as separate mineral species, the nickelian and cobaltian disulfides would represent new minerals. The name bravoite would apply to the intermediate compositions. The boundary between bravoite and the end member in each case is placed at eighty per cent. Although arbitrary, this boundary lies close to established usage in distinguishing between bravoite and pyrite. In the light of this study the pyrite group forms an isostructural series with pyrite FeS_2, vaesite NiS_2 and cattierite CoS_2 as end members, while bravoite $(Co, Ni, Fe)S_2$ is intermediate.

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LATTICE CONSTANTS IN THE PYRITE GROUP

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Abstract

Lattice constants for minerals corresponding to NiS_2 and CoS_2 in the pyrite group are compared with that of pyrite. A cobalt sulphide from the Shinkolobwe mine in the Belgian Congo gives $a_0 = 5.52346 \pm .00048$ Å. A high nickel specimen from the Kasompi mine in the Belgian Congo gives $a_0 = 5.66787 \pm .00008$ Å. Pyrite from Leadville, Colorado, is found to yield the value $a_0 = 5.40667 \pm .00007$ Å.

Through the courtesy of M. Schuiling and M. Vaes of the Union Minière du Haut Katanga, Belgian Congo, two specimens close to the cobalt and nickel end-members of the pyrite group have been made available for study. One specimen was obtained in a core in the process of diamond drilling at the Kasompi mine in Katanga and on chemical analysis has been shown to be close to NiS_2. The other specimen was found in the Shinkolobwe mine near Jadotville in Katanga and has a composition close to CoS_2. Both are described independently, the name *cattierite* being applied to the cobalt mineral and *vaesite* to the nickel mineral. It is the purpose of this paper to record the lattice constants of these natural compounds in comparison with pyrite.

In a study of pyrite and nickelian pyrite Peacock and Smith (1941) determined the lattice constant of nickel-free pyrite from Leadville, Colorado, as $5.4079 \pm .0005$ Å. At the same time pyrite from Rio Marina, Elba, was found to have a lattice constant of $5.4063 \pm .0005$ Å. These lattice constants were based on measurements made on patterns obtained in a Debye camera using iron radiation. Only high angle lines (115), (025), and (125) were used. The Straumanis (1936) method of eliminating errors was employed in obtaining precision lattice constants. At the same time nickelian pyrite from the Denison mine, Sudbury district, Ontario, containing Ni = 6.50 per cent was shown to have a larger lattice constant of $5.419 \pm .002$ Å.

de Jong and Willems (1927) have prepared both NiS_2 and CoS_2 synthetically and have given lattice constants to two decimal places. The figures recorded are: $CoS_2 = 5.64$ Å and $NiS_2 = 5.74$ Å. Previous descriptions of naturally occurring nickelian and cobaltian end-members of the pyrite group do not appear to have been made.

Members of the pyrite group containing the largest amount of nickel which have been previously described are bravoite from Chitina, Alaska, as recorded by Buddington (1924) and bravoite from Mechernich, Prussia, as recorded by Kalb and Meyer (1926). The specimens from both of these localities contain slightly more nickel than iron. In each case, however, the specimen in question falls far short of yielding the percentages necessary to constitute a nickelian end-member. The original bravoite of Hillebrand (1907) was even lower in nickel content and actually contained more iron than nickel.

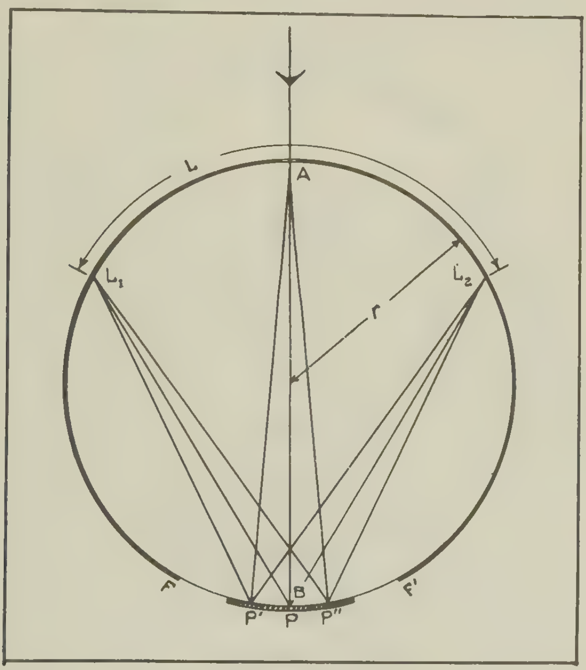

FIG. 1. Schematic diagram of a symmetrical back reflection focussing powder camera (precision camera).

The incident *x*-ray beam enters the camera defined by a single narrow slit on the circumference of the camera at "A." The divergent beam crosses the camera and strikes a considerable area of the sample "B" which is placed on the circumference of the camera diametrically opposite the slit. All particles P', P, P'' in the sample oriented to reflect from a given set of atomic planes (*hkl*) will do so at the same angle and the reflected beams from different portions of the sample will be "focussed" at points "L_1" and "L_2" symmetrically disposed on either side of the center of the film F-F', which is located at the slit "*A*."

Bannister (1940) has studied bravoite from the Mill Close mine in Derbyshire. In the Mill Close specimens more iron was present than nickel although the specimens were substantially nickelian in composition. The work of Bannister suggested the existence of a smooth curve correlating the variation in lattice constant between FeS_2 and NiS_2 with chemical composition.

Lattice constants of three natural specimens essentially end-members of the pyrite group, FeS_2, NiS_2, and CoS_2, have been obtained from x-ray diffraction patterns taken with a back reflection precision focussing camera. The precision determinations have followed the technique of Jette and Foote (1935), and we are indebted to Professor Eric Jette for the camera used in this study. The analytical methods of Cohen (1935) and Jette and Foote (1935) have been employed in order to eliminate systematic errors and provide precision lattice constants for each of the

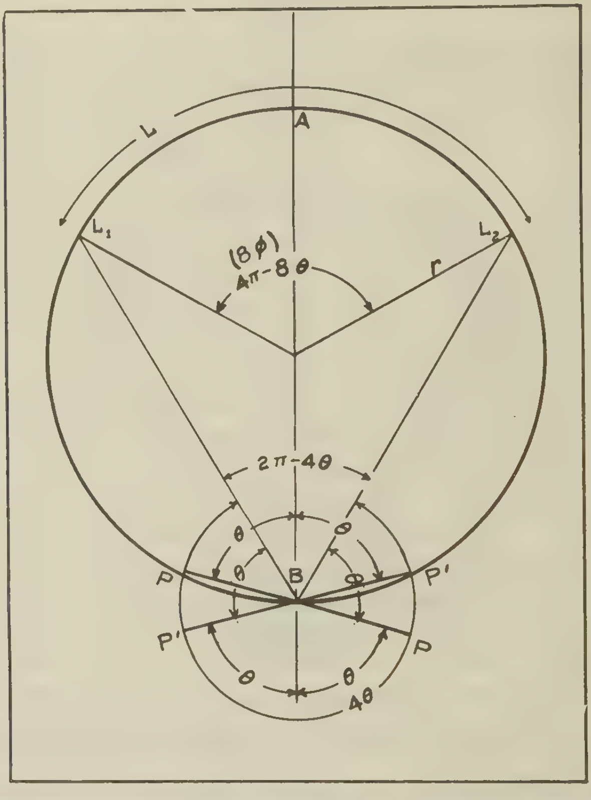

FIG. 2. The geometry of the symmetrical back reflection focussing (precision camera). In terms of the Bragg angle θ and the radius of the camera it is found that the distance "L" separating two corresponding lines on the film is a function of ϕ, the complement of θ rather than θ itself. The distance between corresponding lines "L" in millimeters and the angle ϕ are related by the equation $\phi = L/8R$. The lines (P-P) and (P'-P') represent two orientations of the same set of atomic planes providing the two reflections at points L_1 and L_2 on the film. For simplicity the sample "B" is assumed to be located at a point diametrically opposite the slit "A."

minerals. The method, as used, eliminates systematic errors and, at the same time, handles the random errors due to measurement by a least squares solution which provides the best average representation of the experimental data. The schematic diagram, Fig. 1, will make clear the principle of the symmetrical focussing camera. The geometry of the camera is shown diagrammatically in Fig. 2. The influence of systematic errors due to absorption, film shrinkage, deviation of sample or film from true geometrical position in the camera and other sources of error in lattice constants have been amply treated in the papers of Cohen, and also Jette and Foote.

X-ray patterns were first obtained in a Debye camera using iron radiation in order to index the lines. The lines in a symmetrical focussing precision camera are not indexed directly, since only a few high-angle lines are recorded. The indexing is accomplished by comparing the lines on the precision film with those on a Debye film previously indexed. The distances between corresponding lines "L" on the precision films were measured to hundredths of a millimeter with a microscope and measuring screw calibrated to 0.001 mm. The values were obtained from these measurements (average of four) by means of the equation $\phi = L/8r$ multiplied by the shrinkage correction. The value ϕ instead of θ is used since the separation of corresponding lines in a back reflection camera is a function of ϕ rather than θ in which $\phi = (\pi/2) - \theta$. Using the observed ϕ values, the corresponding $\sin^2\theta$ and $(\phi \sin 2\phi)$ values were obtained from Foote's unpublished tables.* This data and the indices of the lines were then used in setting up the simultaneous equations required by Cohen's method (see references). The solution of these equations for two unknowns (in the case of the isometric system) provides two values A and D. A is the $\sin^2\theta_{100}$ free of systematic errors from which a precise lattice constant a_0 may be obtained by the use of the quadratic form of the Bragg equation, in the case of the isometric system,

$$a_0 = \sqrt{\frac{(\lambda^2/4)(h^2+k^2+l^2)}{A}}.$$

The corrected $\sin^2\theta$ for any line on the film is then found from the equation $\sin^2\theta = A\ (h^2+k^2+l^2)$. The value D called the "drift constant" or constant of proportionality indicates the magnitude of the systematic errors in the observed data.

In order to determine the magnitude of the random errors in the observed $\sin^2\theta$ for each line, a set of theoretical $\sin^2\theta$ values is prepared using the equation $\sin^2\theta = A \cdot (h^2+k^2+l^2)+D(\phi \sin 2\phi)$. Cohen's study showed that the systematic errors in $\sin^2\theta$ were proportional to ($\phi \sin 2\phi$). The difference (Δ) between the observed and theoretical $\sin^2\theta$ is determined and used in a series of computations (see Jette and Foote, 1935) which permit an evaluation of the magnitude of the error (standard error) in the precision lattice constant obtained. Precision lattice constants with a standard error as low as ±.00002 (Jette and Foote, 1935, p. 615) have been obtained under favorable conditions using symmetrical focussing cameras and Cohen's method. The precise lattice constant obtained by the use of Cohen's method is based on an analytical extrapolation to $\sin^2\theta = 100$. It represents within the standard error the best possible lattice constant for the data employed. The "standard error" should be less than ±.0001 for a film with a fair number of sharp lines, as exemplified in the following lattice constants. Pyrite and NiS_2 as shown in Fig. 3 exhibit good resolution, whereas the lines of CoS_2 were somewhat diffused, limiting the accuracy of measurement.

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* Tables were made available through the courtesy of Dr. Frank Foote of the Metallurgical Laboratories of the School of Mines, Columbia University.

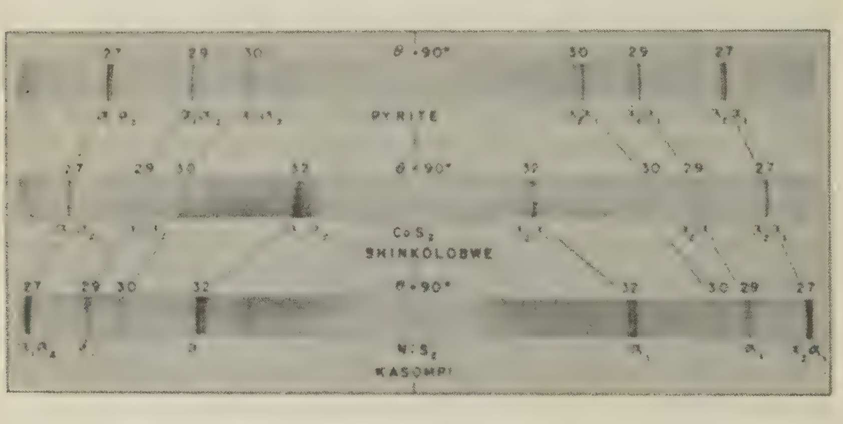

FIG. 3. Precision photographs of natural FeS_2 (pyrite), CoS_2 (cattierite) and NiS_2 (vaesite) as recorded in Table 1. Only the lines recorded are listed as α_1, or α_2. The figures shown above each line are the $(h^2+k^2+l^2)$ values. The relative distances in millimeters between lines $27\alpha_1$, in the three diffraction photographs are 154.27, 175.62 and 196.80.

Pyrite, Leadville	5.40667 ± .00007 Å
NiS_2, Kasompi	6.66787 ± .00008 Å
CoS_2, Shinkolobwe	5.52346 ± .00048 Å

Lattice constants were calculated from the individual lines in each film using the observed $\sin^2\theta$ values and are compared with the precision value obtained by the use of Cohen's method. These a_0 values computed from individual lines show in a striking manner in the pyrite and Kasompi material that the accuracy of the lattice constants determined from individual lines increases with increasing $\sin^2\theta$ value. The same trend is indicated in the less satisfactory Shinkolobwe material.

Table 1

The "a_0" values calculated from individal lines observed in the patterns of pyrite, the cobalt mineral, and the nickel mineral compared with the "a_0" calculated by the use of Cohen's method.

Pyrite

Precision Lattice Constant "a_0" $= 5.40667 \pm .00007$

Shrinkage correction $= \dfrac{130.008}{129.745}$ $1/8R = .00244933$

$(h^2+k^2+l^2)$	(hkl)	"L" mm.	$\sin^2\theta$ Obs.	$\sin^2\theta$ Cor.*	$\Delta \sin^2\theta$	"a_0" obs.	"a_0" adj.*	"d"
$27\alpha_1$	333, 511	154.97	.862199	.862163	$-.000036$	5.40595	5.40605	1.04051
$27\alpha_2$	333, 511	152.90	.862152	.862150	$-.000004$	5.40610	5.40608	1.04051
$29\alpha_1$	520, 432	112.33	.925905	.925925	$+.000020$	5.40642	5.40635	1.00399
$29\alpha_2$	520, 432	109.37	.925892	.925920	$+.000028$	5.40646	5.40637	1.00399
$30\alpha_1$	521	84.27	.957836	.957806	$-.000030$	5.40641	5.40648	.98712
$30\alpha_2$	521	80.24	.957812	.957800	$-.000012$	5.40648	5.40650	.98712

Cobalt Mineral

Precision Lattice Constant "a_0" $= 5.52346 \pm .00048$

Shrinkage correction $= \dfrac{215.01}{214.74}$ $1/8R = .00244933$

$(h^2+k^2+l^2)$	(hkl)	"L" mm.	$\sin^2\theta$ Obs.	$\sin^2\theta$ Cor.*	$\Delta \sin^2\theta$	"a_0" obs.	"a_0" adj.*	"d"
$27\alpha_1$	333, 511	175.62	.825693	.825838	$+.000245$	5.52417	5.52368	1.06299
$27\alpha_2$	333, 511	173.58	.826105	.825839	$-.000266$	5.52279	5.52368	1.06299
$29\alpha_1$	520, 432	139.92	.886804	.887038	$+.000234$	5.52433	5.52360	1.02568
$29\alpha_2$	520, 432	137.39	.887087	.887040	$-.000047$	5.52344	5.52359	1.02568
$32\alpha_1$	440	59.38	.978943	.978842	$-.000101$	5.52319	5.52348	.97642
$32\alpha_2$	440	53.63	.978807	.978843	$+.000036$	5.52358	5.52348	.97642

Nickel Mineral

Precision Lattice Constant "a_0" $= 5.66787 \pm .00008$

Shrinkage correction $= \dfrac{130.008}{129.775}$ $1/8R = .00244933$

$(h^2+k^2+l^2)$	(hkl)	"L" mm.	$\sin^2\theta$ Obs.	$\sin\theta\ ^2$ Cor.*	$\Delta \sin^2\theta$	"a_0" obs.	"a_0" adj.*	"d"
$27\alpha_1$	333, 511	196.80	.784379	.784377	$-.000002$	5.66778	5.66779	1.09078
$27\alpha_2$	333, 511	195.21	.784391	.784376	$-.000015$	5.66774	5.66779	1.09078
$29\alpha_1$	520, 432	166.33	.842472	.842471	$-.000001$	5.66784	5.66782	1.05250
$32\alpha_1$	440	109.78	.929561	.929612	$+.000051$	5.66800	5.66785	1.00159

* Calculated from a_0 and includes systematic error $D(\phi \sin 2\phi)$.

Δ = random errors.

ACKNOWLEDGMENTS

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ECLOGITE INCLUSION FROM THE CAPE PATERSON VOLCANIC NECK IN SOUTH GIPPSLAND, VICTORIA

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ABSTRACT

True eclogite rocks are scarce in Victoria, being known only as occasional fragments that have been carried up to the surface by Tertiary volcanic eruption. The example recorded is of interest because of its simple mineral composition—garnet and omphacite, because of the fresh state of preservation of these primary constituents, and because of the occurrence of *almandine* spinel in the occasional, narrow kelyphitic zones bordering some of the garnet crystals.

INTRODUCTION

Eclogitic rocks have been recorded from many localities in other parts of the world, but detailed descriptions of them are few in number. Only rare occurrences of eclogite occur in Australia, and these are present as inclusions in volcanic necks or pipes. Those on record come principally from New South Wales; one or two have been referred to in Victoria.

E. F. Pittman (11) described volcanic breccia from Ruby Hill, Bingera in N.S.W., as consisting chiefly of angular fragments with occasional rounded pebbles of various rocks, included among which was eclogite. He also recorded lumps of eclogite from the supposed diamond-bearing volcanic pipe at Snodgrass, near Delegate in N.S.W., in material which has the characters of a volcanic breccia. Eclogite inclusions from elsewhere in New South Wales were recorded from Bundundah, from ten miles east of Warialda, from 3 miles north of Allyn Brook, and from four and a half miles north of Kandie Peak (12).

G. W. Card (3) described the eclogite from Ruby Hill, Bingera, as being abundant in pieces of all sizes and as being readily recognised by its constituent garnet and pyroxene. Card (4) also placed on record the occurrence of eclogite from Mullimbibby, N.S.W.

H. J. Grayson and D. J. Mahony (9) described an ejected block of pyroxenite from the Western District of Victoria as consisting of dark green pyroxene and pale pink garnets. They regarded this rock as being allied to eclogite. An additional occurrence of eclogite in Victoria occurs among the collection of rocks at the Victorian Mines Department Geological Museum. This rock (section no. 2234 and sample no. 7593 in the Mines Dept. Collection) is referred to as eclogite, labelled as coming from Benambra, and said to be from a pipe on the Snowy River in the Benambra District. It is described as a coarse-grained mixture of pyroxene, amphibole and garnet. Another example in this collection (section no. 690) is labelled "Basic Concretion?" from the Old Landing Place, Cape Paterson. It is a rock type comparable with the eclogites, consisting of greenish-brown garnet, pyroxene and an indeterminate, brownish coloured alteration product. It is one of the "basic concretions" referred to by A. E. Kitson (10) in his work on the Volcanic Necks from South Gippsland.

The eclogite from the volcanic neck at Cape Paterson, South Gippsland, is apparently the first record of true eclogite from such a source in Victoria. Only one small specimen, weighing approximately 10 grams before sectioning, was found at Cape Paterson. It was collected by Mr. C. Norris, Chief Surveyor at the State Coal Mine, Wonthaggi. The volcanic neck from which the eclogite specimen was obtained, has been described in some detail by A. E. Kitson (10), W. F. Ferguson (7) and A. B. Edwards (6).

The neck is a pear-shaped outcrop of ¾ acre in extent that intrudes Jurassic sediments. Kitson (10) referred to pebbles contained in the neck as having the same general shape as those in the adjacent Jurassic rocks, and as being broken in a similar way. Ferguson (7) showed, on a plan of the neck (7, plate VI), the positions of coarse agglomerate and tuff, dense basalt, sandstone, mudstone, masses of olivine, indurated shale and ferruginous veins. Edwards stated (6) that the greater part of the plug was occupied by a dense, blue-black tuff composed of fragments of basic igneous rock. Inclusions of Jurassic rock were recorded as numerous. A dunite xenolith in roughly columnar monchiquite found in parts of the neck, was noted as consisting almost entirely of olivine and picotite.

DESCRIPTION OF THE CAPE PATERSON ECLOGITE

Prior to sectioning, the specimen was a small, rounded nodular sample with an irregular surface. It is a fresh basic rock having a green coloured base of omphacite, and is studded with pink to deep reddish coloured grains of garnet. Chemical work on eclogites from other localities where samples were abundant enough for analytical purposes, has shown that they have compositions like basic igneous rocks or basic derivatives of magmas, mainly gabbros. Some have chemical affinities with peridotites and pyroxenites (2).

The chief interest in the Victorian specimen centres around the fact that it is a true eclogite of simple mineral composition, conforming with A. R. Alderman's statement (1) that the term "eclogite" should be used to denote a rock consisting essentially of omphacite and garnet, the garnet having the almandite and pyrope molecules generally dominant, in agreement with Haüy's original definition.

Examples of eclogite from other parts of the world, contain in addition such primary minerals as cyanite, hornblende, glaucophane, various kinds of mica, epidote, zoisite, olivine, clinozoisite and rhombic pyroxene; also accessory quartz, rutile, ilmenite, apatite and pyrrhotite. Perhaps these are not seen in the Cape Paterson eclogite, because of the small amount of rock available, which may not therefore be entirely representative of the eclogite as it occurs in depth.

The rock has a granular texture, is medium grained and tends to be inequigranular with a few of the grains of garnet ranging up to 7 mm. across, and so forming occasional porphyritic individuals. The specific gravity of the rock is 3.31.

The essential minerals, consisting of primary pink garnet belonging to the almandite—pyrope group, and green augite of the variety known as omphacite, are of xenoblastic habit, the garnets being granular and rarely showing crystal faces. Numerous irregular cracks traverse the grains of the garnet (Fig. 1). The omphacite, which is bright green in the hand specimen, is colourless to pale green in thin section, with occasional well-marked cleavages, extinction angles up to 41°, R.I. = 1.675, 2V variable from 60° to 80°, and optically positive. It shows an occasional tendency towards the development of crystal faces and is mainly of smaller grain size than the garnet. Some of the omphacite grains measure as much as 4 mm. in length.

The only accessory mineral present is the violet coloured variety of spinel known as *almandine* ($MgO.Al_2O_3$). It occurs as small, irregular crystals, seldom displaying crystal faces and commonly appearing in the nature of angular fragments. Spinel is apparently a rare mineral in eclogites; G. W. Card refers to a yellowish-brown spinel with a broad black margin, a variety which he determined as pleonaste, in the eclogite from Ruby Hill, Bingera. Apart from this, the author has seen no other record of the occurrence of spinel in eclogites.

Secondary minerals are uncommon in the eclogite from Cape Paterson, principally because of the freshness of the rock. Narrow, brownish-green, kelyphitic reaction borders, as in the Mullimbibby (4) and Ruby Hill (3) eclogites from New South Wales, and other extra-Australian examples (1 and 2), occur in places around several of the garnet grains, and appear to consist of fibrous hornblende with a very small amount of basic plagioclase and occasional grains of diopside; these constituent minerals in the kelyphitic borders, are of microscopic dimensions.

The violet coloured, microscopically small spinel grains are mainly confined to the outer portions of the alteration zones (Fig. 1), but a few occur enclosed in the omphacite; even in the omphacite, however, they are always very close to the reaction borders of the garnets.

The association of spinel with the reaction zones in this rock, suggests development by the same process which formed the kelyphitic zones. MgO would be available from the pyrope molecule in the garnet, while sufficient Al_2O_3 would be available from both the almandite and pyrope molecules. The FeO present would go to form the small amounts of hornblende in the kelyphitic borders. Lime for the production of minute amounts of the basic feldspar would be derived partly from the garnet and partly from the omphacite, as the garnet constituent of eclogites is usually, to use Fermor's terminology (8), "a calc-pyralmandite variety with 70% to 86% of pyrope and almandite, the remainder being lime garnet."

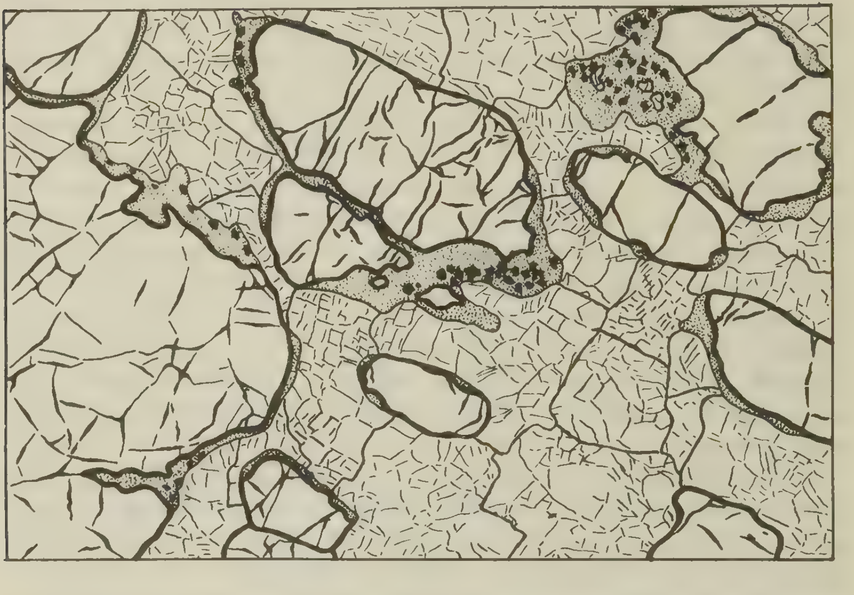

FIG. 1. Sketch of micro-section of eclogite (×9) showing garnet with kelyphitic reaction zones. Opaque areas represent *almandine spinel* in the outer regions of the reaction borders. Omphacite forms the groundmass.

J. A. Dunn (5) has also described the development of occasional examples of spinel within and close to garnet, by reaction in a garnet-cordierite gneiss from Mogok in India. Dunn pictured the garnet as supplying some of the constituents for the reaction, and he regarded garnet molecules as diffusing through, or being carried by, cordierite to sillimanite where reaction produced a secondary intergrowth of spinel and cordierite. According to Dunn, the presence of cordierite was necessary before the action could take place, but in the reaction zones of the garnet in the eclogite from Cape Paterson, there is no evidence that either cordierite or sillimanite played any part in the reaction which produced spinel.

The occurrence of the kelyphitic rims of plagioclase and hornblende around garnets, is regarded by Alderman (2) as developing at quite an early stage in the retrograde changes undergone by eclogites. The dark rings thus formed around the garnet are attributed to reaction between iron-rich garnet and omphacite (1). The changes in the Cape Paterson eclogite seem to represent an even earlier stage of alteration, as some of the garnets are entirely unaffected, while those with the alteration rims only have incomplete kelyphitic borders; in addition, the omphacite is entirely unaffected, being quite fresh and clear. Nevertheless, the evidence, however slight, exists to show that retrogressive mineral change had begun.

The small inclusion of eclogite from the Cape Paterson volcanic neck, is not necessarily co-magmatic with the basaltic material of the volcanic neck. It was most probably derived from a deep-seated source of consolidated basic rock, and was torn off from the walls of the vent up which the basaltic lava rose; it was carried up to the surface among other types of country rock gathered up during the ascent of the lava. At the surface, the eclogite has become subject to minor amounts of retrogressive mineral changes, consequent upon the eclogite-minerals becoming unstable on arriving in regions of lower temperature and lower pressure, where they are out of harmony with the changed physical conditions. The minerals retained their original form by virtue of the rapid process, i.e. volcanic eruption and explosion, by which they were carried up from regions of high temperature and high pressure.

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MONTMORILLONITE

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ABSTRACT

Montmorillonite (including nontronite) probably varies in composition between the following end-member formulas: $4(Al, Fe)_2O_3 \cdot 16SiO_2 \cdot 20H_2O$, $6(Al, Fe)_2O_3 \cdot 12SiO_2 \cdot 22H_2O$, $6MgO \cdot 3(Al, Fe)_2O_3 \cdot 12SiO_2 \cdot 25H_2O$, and $6MgO \cdot 2(Al, Fe)_2O_3 \cdot 14SiO_2 \cdot 24H_2O$. These variations are shown on a (partial) triangular prism on which 52 analyses are plotted. The optical properties vary very little except with variations in the Al-Fe''' series.

INTRODUCTION

In 1935 Marshall,[1] gave good evidence for the conclusion that montmorillonite is essentially $Al_4(OH)_{12}Si_8O_{12}(OH)_8$, in which Si may be replaced (in part) by Al, and Al may be replaced (in part) by Mg. To a less extent he concluded that Si may be replaced by P and perhaps by Fe''' and Ti; also Al may be replaced entirely by Fe''', or in part by Ti, Fe'', Mn, Ca, and Na.[2] He also showed that structurally montmorillonite consists of repetitions of one $Al_4(OH)_{12}$ layer and two $Si_4O_6(OH)_4$ layers. In this respect it differs from kaolinite which consists of repetitions of one $Al_4O_4(OH)_8$ layer and one Si_4O_6 layer.

DERIVATION OF END-MEMBER FORMULAS

If the formula of montmorillonite be doubled, for convenience, it becomes $Al_8(OH)_{24}Si_{16}O_{24}(OH)_{16}$ or $4Al_2O_3 \cdot 16SiO_2 \cdot 20H_2O$. This may be accepted as one end-member of the montmorillonite system. Since Si may be replaced (in part) by Al, Si_{16} might become $Si_{15}Al$, $Si_{14}Al_2$, $Si_{13}Al_3$, etc. If the uneven numbers are omitted (for convenience in writing the oxide formulas) the series becomes: Si_{16}, $Si_{14}Al_2$, $Si_{12}Al_4$, $Si_{10}Al_6$, and corresponding oxide formulas are $4Al_2O_3 \cdot 16SiO_2 \cdot 20H_2O$, $5Al_2O_3 \cdot 14SiO_2 \cdot 21H_2O$, $6Al_2O_3 \cdot 12SiO_2 \cdot 22H_2O$, and $7Al_2O_3 \cdot 10SiO_2 \cdot 23H_2O$ (the number of H_2O molecules being assumed to be such as will keep the total number of oxygen atoms constant). It is at present unknown how far this replacement of Si by Al can proceed; two analyses of natural minerals (1 and 2) give the ratio: $7Al_2O_3 . 10SiO_2$ almost exactly and a few others have a little more than $6Al_2O_3$ to $12SiO_2$, but since analyses of these minerals are almost inevitably made on clay-like masses rather than on single crystals or aggregates of crystalline material of one kind, it seems probable that the material analyzed may include some bauxite or limonite in certain cases.

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[1] *Zeit. Krist.*, **91**, 433 (1935).

[2] Noll (*Chem. Erde*, **10**, p. 129) has shown that Be may be present in large amount. One per cent of CuO is present in analysis 35. Analyses 33, 39, and 51 are K-montmorillonites.

There is little doubt that the replacement goes at least to $6Al_2O_3 \cdot 12SiO_2$; it will be assumed that this is a second end-member.

Considering next the replacement of Al by Mg, Noll[3] has proved that montmorillonite can be made artificially containing as much as 15.30 per cent of MgO. This is considerably more than has ever been found in any natural mineral and may reasonably be taken as the maximum that is possible. Noll's analysis may be expressed as $6MgO \cdot 3Al_2O_3 \cdot 12SiO_2 \cdot 20H_2O$. As compared with the formula, $6Al_2O_3 \cdot 12SiO_2$, obtained above, this shows a replacement of just half the Al atoms by Mg atoms. If all the aluminum could be replaced by magnesium, the formula would be $12MgO . 12SiO_2$ (with H_2O). It seems impossible that this is an end-member of montmorillonite, but it is convenient to use it as if it were. Then it is possible (disregarding other variations) to represent montmorillonite analyses on a triangle having $4Al_2O_3 \cdot 16SiO_2$, $6Al_2O_3 \cdot 12SiO_2$, and $12MgO \cdot 12SiO_2$ at its corners, as in Fig. 1. Fifty-two analyses of montmorillonite (including beidellite, bentonite, leverrierite, and nontronite[4]) have been

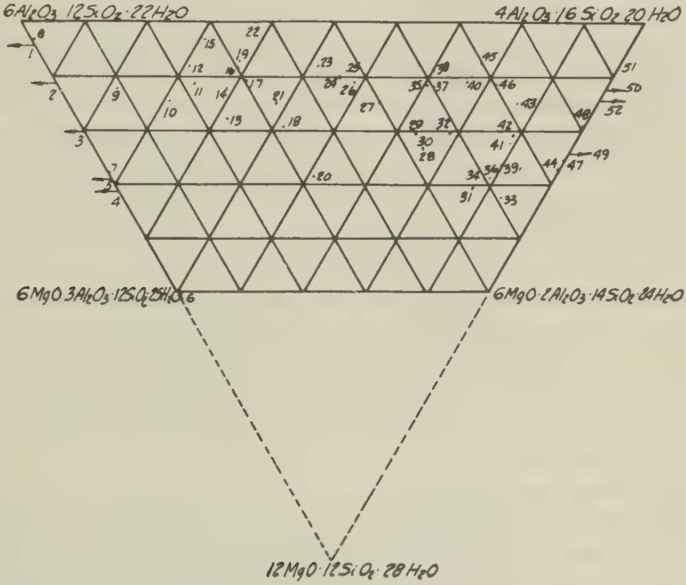

FIG. 1. Montmorillonite analyses plotted on (part of) a triangle having $4Al_2O_3 \cdot 16SiO_2 \cdot 20H_2O$, $6Al_2O_3 \cdot 12SiO_2 \cdot 22H_2O$, and $12MgO \cdot 12SiO_2 \cdot 28H_2O$ at its corners, Al standing for Al, Fe‴, Ti and Cr, and Mg for Mg, Fe″, Mn, Ca.

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[3] *Chem. Erde*, **10**, 129 (1936).

[4] Chemically, attapulgite belongs in this system, but it seems to differ in crystal structure, as shown by de Lapparent, J. (*Bull. Soc. Fr. Min.*, **61** p. 253) and Bradley, W. F.: *Am. Mineral.*, **25**, 405 (1940).

plotted on the diagram; the distribution of the points representing these analyses indicates that the probable end-member formulas are:

1. $4Al_2O_3 \cdot 16SiO_2 \cdot 20H_2O$
2. $6Al_2O_3 \cdot 12SiO_2 \cdot 22H_2O$
3. $6MgO \cdot 3Al_2O_3 \cdot 12SiO_2 \cdot 25H_2O$
4. $6MgO \cdot 2Al_2O_3 \cdot 14SiO_2 \cdot 24H_2O$

The writer would suggest that the first end-member should be designated leverrierite. Marshall states that the chief replacement in beidellite is Al for Si; accordingly the second formula is apparently the end-member most fittingly called beidellite.

As indicated by the arrows along the margins of the drawing a few analyses contain an excess of Al_2O_3 (due to admixed bauxite?) and a few contain an excess of SiO_2 (due to admixed quartz or cristobalite?). Of course, these analyses suggest the possibility of a greater replacement of Si by Al, and *vice versa*, than recognized in the drawing, but if more Al_2O_3 were present it is difficult to understand why kaolinite would not form instead of montmorillonite.

In calculating the position of each analysis on Fig. 1, Al is assumed to include Fe''' (and Ti and Cr, if they are present). Also Mg is assumed to include Fe'', Mn, and Ca. Alkalies have been disregarded entirely; they are present in such small quantity (except in analyses 33, 39, and 51) that the resulting error is not important; K is important in analyses 33, 39, and 51, but, as noted by Ross and Shannon,[5] it seems to replace H, or H_2O, rather than Mg or Al.

The analyses show that Al may be replaced by Fe''' in any amount from 0 to 100 per cent. To show this graphically, a three-dimensional figure is necessary, as provided in Fig. 2, in which the vertical axis represents $100\ (Fe'''+Ti+Cr)$ divided by $Fe'''+Ti+Cr+Al$. The iron end-member formulas are:

1. $4Fe_2O_3 \cdot 16SiO_2 \cdot 20H_2O$
2. $6Fe_2O_3 \cdot 12SiO_2 \cdot 22H_2O$
3. $6MgO \cdot 3Fe_2O_3 \cdot 12SiO_2 \cdot 25H_2O$
4. $6MgO \cdot 2Fe_2O_3 \cdot 14SiO_2 \cdot 24H_2O$

These are the probable limits of variation in composition of the mineral commonly known as nontronite, but this is merely the ferric iron end of the montmorillonite system. Analysis 45 is the original chloropal or unghwarite. Accordingly it may be reasonable to designate $4Fe_2O_3 \cdot 16SiO_2 \cdot 20H_2O$ as chloropal.

The tenor of H_2O in montmorillonite varies notably, apparently good analyses ranging all the way from 15 to 25 per cent, but the usual amount is about as given in the formulas; though the evidence that some end members contain more than others is not conclusive. Formulas are written with varying tenor of water so as to keep the total number of oxygen atoms constant.

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[5] *Jour. Am. Ceram. Soc.*, **9**, 77 (1926).

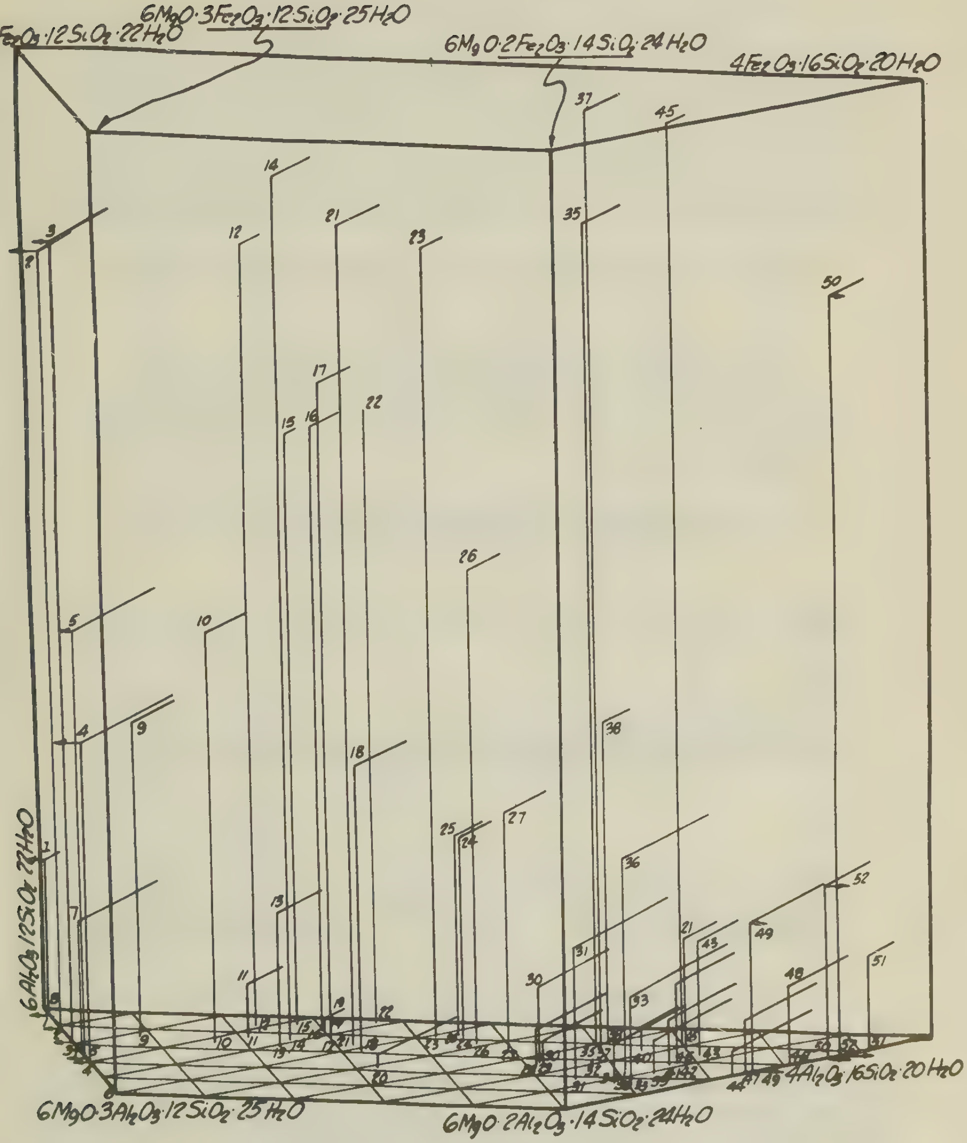

Fig. 2. Montmorillonite analyses in a three-dimensional figure whose base is shown in Fig. 1.

OPTIC PROPERTIES AS RELATED TO COMPOSITION

It is well known that the indices of refraction of montmorillonite (including nontronite) vary considerably with variations in the tenor of water. For example, Larsen and Steiger[6] show that nontronite from Woody, California, with 13.06% H_2O at ordinary temperature has an index of 1.585, which rises to 1.615 at 75°C. with 3.40% H_2O and to 1.67 at 210°C. with 1.80% H_2O. Other writers have noted similar data for ordinary montmorillonite. For example, Kerr[7] states that the indices of refraction of montmorillonite from France and from Styria rise appreciably even at ordinary temperature if allowed to stand in a desiccator. Such variations probably explain the fact that Kerr reports the indices of the mineral from France as 1.506 and 1.485 while Ross and Shannon[8] report the indices of the same mineral of the same composition as 1.527 and 1.504, and also Larsen and Wherry[9] report that the indices of mont-

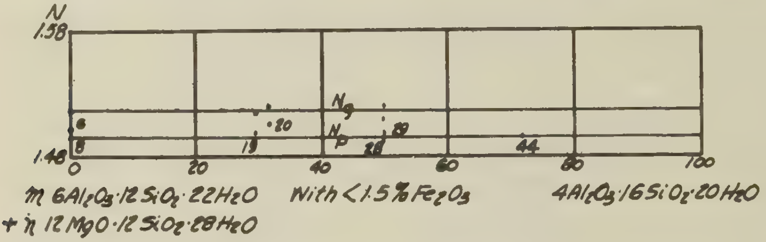

FIG. 3. Indices of refraction of montmorillonites (containing less than 1.5% Fe_2O_3) with varying replacement of Si by Al.

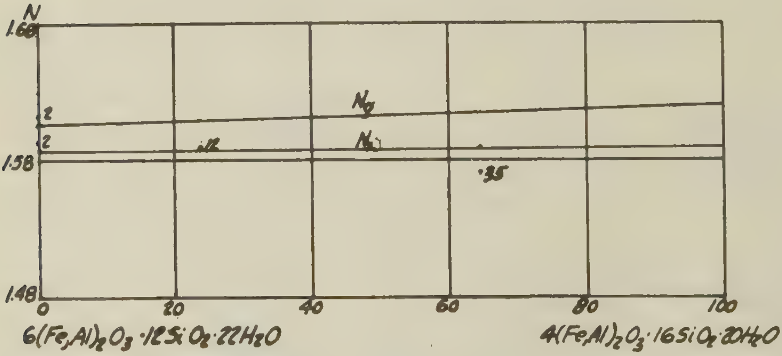

FIG. 4. Indices of refraction of montmorillonites (or nontronites) with much Fe_2O_3 (between 10 and 13% atomic Al).

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[6] *Am. Jour. Sci.*, **15**, 1 (1928).

[7] *Am. Mineral.*, **17**, 192 (1932).

[8] *Jour. Am. Cer. Soc.*, **9**, 77 (1926).

[9] *Jour. Wash. Acad. Sci.*, **7**, 208 (1917).

morillonite from Beidell, Colorado, increase in immersion liquids from 1.575 and 1.470 to 1.602 and 1.558, while Ross and Shannon give the indices of this same mineral as 1.536 and 1.494. It is probably due to these facts that it seems to be impossible to find the exact relations between variations in composition and in optical properties.

But careful study of the data shows that replacement of Si by Al has very little effect upon the optical properties. This is shown graphically in Figs. 3 and 4. All reliable data on montmorillonite containing very little iron ($100Fe'''/Fe'''+Al<4$) are included in Fig. 3; all data for samples with much iron ($100Fe'''/Fe'''+Al=80–87$) except 21 and 23 (excluded on account of unsatisfactory optical data) are included in Fig. 4. Replacement of Al by Mg also has very little effect on the optical properties; this is well shown by the similarity in properties between samples 8 and 6, as well as by Fig. 5.

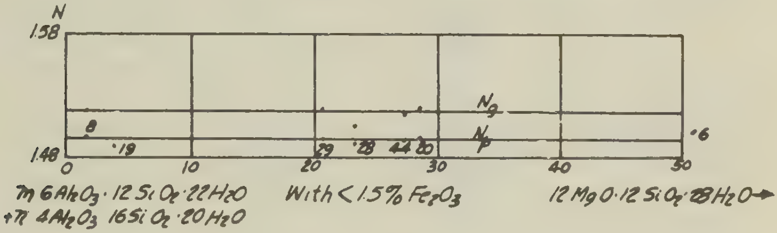

FIG. 5. Indices of refraction of montmorillonites (containing less than 1.5% Fe_2O_3) with varying replacement of Al by Mg.

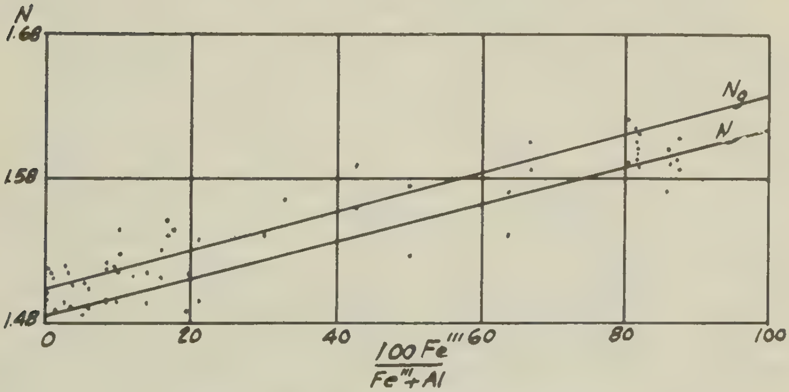

FIG. 6. Indices of refraction of montmorillonites with varying replacement of Al by Fe.

On the other hand replacement of Al by Fe produces a marked effect on the optical properties, as shown on Fig. 6, which is based solely on this relation, because other replacements have no important effect. It is probable that variations from the rectilinear relationship are due in large part to variations in tenor of water.

TABLE 1. DATA USED IN FIGS. 1–6

	Si atoms for 24 non O(+H) atoms	$6Al_2O_3 \cdot 12SiO_2 \cdot 22H_2O$	$12MgO \cdot 12SiO_2 \cdot 28H_2O$	$4Al_2O_3 \cdot 16SiO_2 \cdot 20H_2O$	$\frac{100(Fe'''+Cr+Ti)}{Fe'''+Cr+Ti+Al}$	Sign	2V	N_g	N_m	N_p	N_g-N_p	G
1.	10.29	96.0	4.0	—	16.9			1.550		1.540	.01	
2.	10.30	88.4	11.6	—	81.4	+	Sm.	1.612	1.604	1.592	.02	2.296
3.	11.18	79.7	20.3	—	87.4	−	26°	1.608	1.593	1.585	.023	2.289
4.	11.23	68.6	31.4	—	33.2	−	30°	1.585	1.585	1.559	.026	
5.	11.34	71.3	28.7	—	32.9				1.564			
6.	11.94	48.9	51.1	—	0.0				1.50			
7.	11.99	72.1	27.9	—	14.2			1.588		1.569	.019	
8.	12.00	96.3	3.7	—	0.3			1.518		1.495	.023	
9.	12.35	78.4	12.9	8.7	23.4	—	0°	1.578	1.578	1.538	.04	2.498
10.	12.70	69.4	14.5	16.0	42.9	−	30°	1.588	1.588	1.559	.029	
11.	12.84	67.1	11.7	21.2	5.9	−	16°	1.549		1.517	.032	
12.	12.90	69.5	8.1	22.4	81.9	+	40°	1.610	1.600	1.589	.021	2.271
13.	12.92	58.3	18.0	23.7	17.9				1.544			2.40
14.	13.03	61.1	12.5	26.3	90.5	−	Sm.	1.573		1.569	.004	
15.	13.10	69.3	3.1	27.6	61.4							
16.	13.19	61.3	9.0	29.7	63.5	−	29°					
17.	13.22	58.8	11.2	30.0	69.0			1.605		1.585	.020	
18.	13.26	48.4	19.7	31.9	30.3				1.542			
19.	13.29	60.0	7.8	30.2	1.4			1.513		1.488	.025	
20.	13.30	39.5	28.2	32.3	0.9	−		1.517		1.494	.023	
21.	13.31	52.1	15.1	32.7	86.7	−	Sm.	1.575		1.571	.004	
22.	13.48	62.9	—	37.1	63.9			1.57		1.54	.03	
23.	13.66	48.8	8.0	43.2	83.8				1.57–1.63			
24.	13.83	44.3	10.1	45.6	21.0			1.536		1.494	.042	
25.	13.87	43.5	9.7	46.8	21.2		Sm.	1.536		1.494	.042	
26.	13.90	40.1	11.2	47.7	50.2			1.572		1.523	.049	
27.	13.98	35.8	14.4	49.8	22.8							
28.	14.07	24.3	23.5	52.2	3.9			1.506		1.490	.016	
29.	14.11	26.6	20.8	52.5	2.8			1.520		1.495	.025	
30.	14.14	24.1	23.2	52.7	8.3				1.520		.007	
31.	14.22	11.6	31.0	57.4	14.0		16–24°	1.513		1.492	.021	
32.	14.23	20.9	20.8	58.3	0.0							
33.	14.27	6.7	32.7	60.6	9.3	−	12–25°	1.565		1.543	.021	
34.	14.39	11.6	28.9	59.5	3.8				1.480			1.98
35.	14.40	29.1	11.3	59.6	86.0	−	66°	1.60	1.59	1.57	.03	
36.	14.40	10.3	29.0	60.7	23.2				1.546–1.553			2.03
37.	14.41	27.9	12.0	60.1	98.2	−	Sm.	1.585	1.585	1.57	.015	2.495
38.	14.46	29.6	8.9	61.5	34.0							
39.	14.52	9.9	26.8	63.3	3.7	−	15–27°	1.550		1.528	.022	
40.	14.56	24.5	10.9	64.6	1.7							
41.	14.69	10.2	22.9	66.9	5.4	−	13–24°	1.508		1.484	.024	
42.	14.73	10.5	21.5	68.0	9.1	−	7–19°	1.515		1.493	.022	
43.	14.74	13.3	15.3	71.4	12.3				1.558			
44.	14.78	—	27.3	72.7	3.1		16–24°	1.513		1.492	.021	
45.	14.80	21.6	8.2	70.2	96.7							
46.	14.80	18.6	11.7	69.7	11.8				1.51±			
47.	14.96	—	25.9	74.1	5.8			1.506		1.490	.016	
48.	15.27	—	19.3	80.7	8.5	−	15–22°	1.515		1.492	.023	
49.	15.49	—	24.0	76.0	15.9	−		1.53		1.51	.02	
50.	15.74	—	12.4	87.6	80.5			1.62		1.59	.03	2.29
51.	15.86	—	8.6	91.4	10.3			1.545		1.525	.02±	
52.	16.52	—	14.6	85.4	19.9	−	18°	1.514		1.487	.027	

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THE CRYSTAL STRUCTURE OF α-SIC, TYPE VI

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Abstract

A structure has been determined for α-SiC, type VI, one of the new modifications of SiC recently reported by Thibault. He found the rhombohedral unit cell to contain 11 SiC, with the dimensions $a_{\text{rh}}=27.704$Å and $\alpha=6°21.5'$. Space group *R3m*. Referred to hexagonal axes, the unit cell contains 33 SiC, with $a=3.073$Å and $c=82.94$Å. Following a procedure similar to that used for type IV, the following atomic positions have been determined for type VI:

Hexagonal unit cell—
11 Si at 000, $0\ 0\ 2z$, $0\ 0\ 6z$, $0\ 0\ 8z$, $0\ 0\ 12z$, $0\ 0\ 15z$, $0\ 0\ 18z$, $0\ 0\ 21z$, $0\ 0\ 25z$, $0\ 0\ 27z$, $0\ 0\ 31z$.
11 C at $00p$, $0\ 0\ 2z+p$, $0\ 0\ 6z+p$, $0\ 0\ 8z+p$, $0\ 0\ 12z+p$, $0\ 0\ 15z+p$, $0\ 0\ 18z+p$, $0\ 0\ 21z+p$, $0\ 0\ 25z+p$, $0\ 0\ 27z+p$, $0\ 0\ 31z+p$.
11 Si and 11 C at 2/3 1/3 1/3 plus the above coordinates.
11 Si and 11 C at 1/3 2/3 2/3 plus the above coordinates.
$z=1/33$; $p=1/44$

In his investigation of silicon carbide, Thibault[1] discovered two new rhombohedral modifications of α-SiC, which he designated as types IV and VI. This brought to four the number of known types based on rhombohedral unit cells, namely, type I with 5 SiC in the unit cell, type IV with 7 SiC, type VI with 11 SiC, and type V with 17 SiC. Ott determined the structure of type I[2] and reported the existence of type V[3], but made no attempt at a structure determination of the latter. The author recently published an account of the structure of type IV,[4] and the present paper deals with the structure of type VI. The structure of type V has also been determined, and an account of this will appear in the near future.

In these structure determinations both Ott and the writer have made the assumption that in all of the types there is a common structural arrangement in which each silicon atom is surrounded tetrahedrally by four carbon atoms, and each carbon by four silicon. This assumption seems justified on two grounds; first, the very excellent agreement between the observed intensities and those calculated for such a tetrahedral arrangement, and second, the very close crystallographic relationships between the various types. The vertical heights of all of the unit cells are integral multiples of a common unit, while the horizontal dimensions remain constant. Certain forms are common to two or more types. *X*-ray photographs of the various types have certain reflections in common. All of these facts seem to point indisputably to a common basic structure. If this is true, then in each type all atoms are located on the vertical symmetry axes, and the types differ merely in the distribution of atoms along these axes. This relationship may be expressed in another way. All of the structures are built up of equally spaced, identical layers. The various types differ only in the relative horizontal displacement of these layers. There are three possible positions for a single layer, and the sequence in which these positions are successively occupied constitutes the distinguishing feature of each type.

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[1] Thibault, N. W., Morphological and structural crystallography and optical properties of silicon carbide: *Am. Mineral.*, **29**, 249–278, 327–362 (1944).

[2] Ott, H., Das Gitter des Karborunds (SiC): *Zeits. Krist.*, **62**, 291–317 (1925).

[3] Ott, H., Eine neue Modifikation des Karborunds: *Probleme der modernen Physik.* Arnold Sommerfeld Festschrift. S. Hirzel, Leipzig, (1928).

[4] Ramsdell, L. S., The crystal structure of α-SiC, type IV: *Am. Mineral.*, **29**, 431–442 (1944).

In this investigation of the structure of type VI the tetrahedral arrangement has been taken for granted. However, even with this assumption the solution would still remain very difficult. As the size of the unit cell increases, the number of geometrically possible arrrangements increases very rapidly. For type I, Ott had to choose between two possible sequences. For type IV, only five arrangements had to be considered. But for type VI, there are 42 different arrangements geometrically possible, while for type V the number is in the thousands. Unless some additional simplifying assumption is made, the task of choosing among the various possibilities would be very great.

In all of the modifications whose structures are known, both in the hexagonal and the rhombohedral types (I, II, III and IV) the arrangements of silicon atoms along the vertical symmetry axes are such that adjacent atoms are separated by intervals of two, three and four layers only. Ott recognized this apparent limitation, and referred to it as the "Minimalprinzip." If it is assumed that this limitation holds for type VI, the number of possible arrangements is at once reduced from 42 to 5. The writer does not know why such a limitation might exist, but there seems to be nothing unreasonable about it. The justification for applying the limitation to type VI lies in the agreement between the observed intensities and those calculated for the proposed structure. This agreement is so excellent that it hardly seems possible that it could be due to chance, and that some more complex arrangement is the correct one.

The method used in determining the geometrical possibilities for type VI is similar to that used for types I and IV, and can be most easily described in terms of the hexagonal unit cell. This hexagonal unit is 33 layers high, with 11 silicon atoms on each of the three vertical symmetry axes, which pass through the points 000, 2/3 1/3 0 and 1/3 2/3 0. These three axes will be referred to as *A*, *B*, and *C*, respectively. Because of the tetrahedral arrangement, only one of the three axes may be occupied at any given level, and no axis may be occupied at two adjacent levels. Moreover, since the lattice is actually rhombohedral, the sequence of atoms along A, beginning at 000, must be identical with the sequence along B, beginning at 2/3 1/3 1/3, and with that along C, beginning at 1/3 2/3 2/3. This displacement of 1/3 and 2/3 along B and C, relative to A, means that for any silicon atom on A at the nth level, there will also have to be silicon atoms at $n+11$ and $n+22$ on B and C, respectively. Hence no two silicon atoms on A, B or C can be 11 or 22 layers apart. These restrictions are automatically taken care of by use of the following tabulation:

0	(1)	2	3	4	5	6	7	8	9	10
(11)	12	13	14	15	16	17	18	19	20	21
(22)	23	24	25	26	27	28	29	30	31	(32)

Permissible sequences will consist of 11 numbers, one to be chosen from each vertical column. Two cannot be chosen from the same vertical column because they are separated by intervals of 11 or 22. No two adjacent horizontal numbers may be chosen. Accordingly, if the sequence starts with 0, the numbers 11, 22, 1 and 32 may not be used. A possible sequence would thus be 0, 12, 2, 14, 4, 16, 6, 18, 8, 20, 10, which arranged in increasing order would become 0, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, 0. The intervals between the successive numbers are 2-2-2-2-2-2-2-2-2-2-13. It is the intervals between the numbers which are characteristic. The particular number with which a sequence starts is of no importance. There are 681 permissible sequences which can be derived from the tabulation, but when expressed as intervals, there are found to be only 42 different sets. These vary from the example given above, with a maximum interval of 13, down to five arrangements involving only intervals of 2, 3 and 4. These five arrangements are as follows:

(1) 32433333423
(2) 33432323433
(3) 22444234332
(4) 23343334332
(5) 24243334242

The theoretical intensities to be expected from each of these five arrangements have been calculated for a sufficient number of reflecting planes to determine whether or not there is any significant correlation with the actual observed intensities for type VI. The first four arrangements show marked discrepancies between observed and calculated intensities, and are accordingly discarded. The fifth arrangement shows excellent agreement, and is accepted as the correct one.

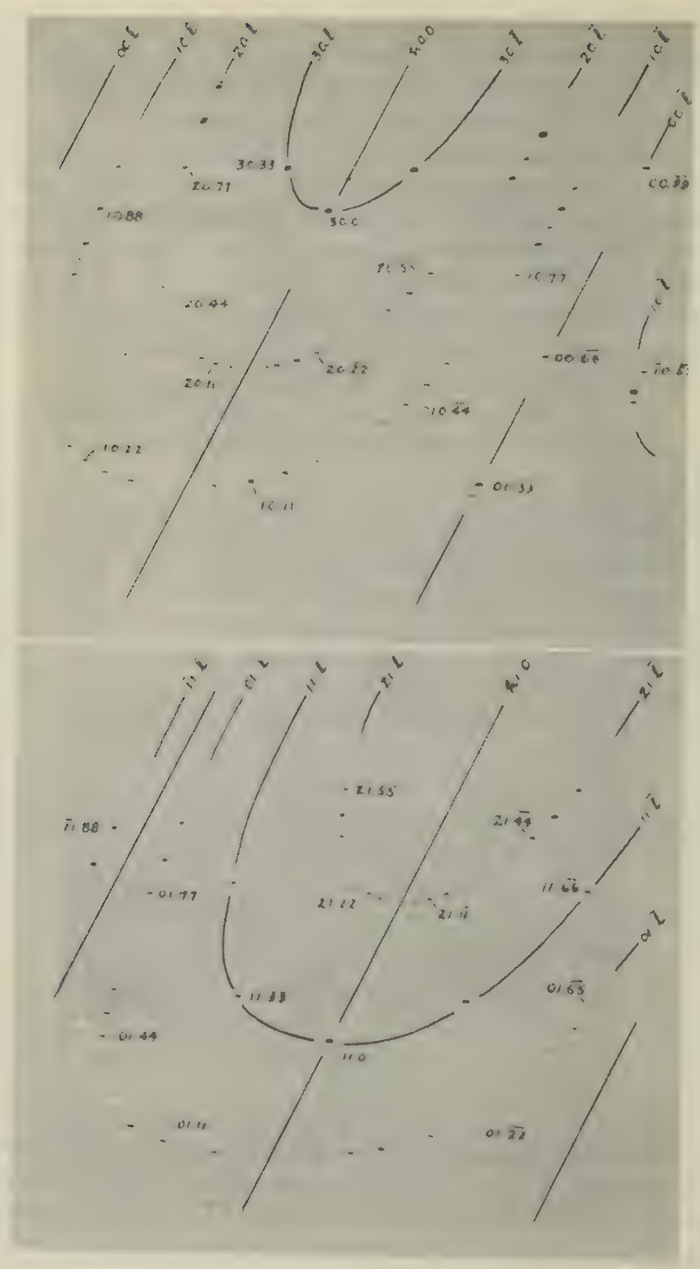

FIG. 1. Weissenberg photographs of α-SiC, type VI; a-axis rotation. Above—zero level; below—first level.

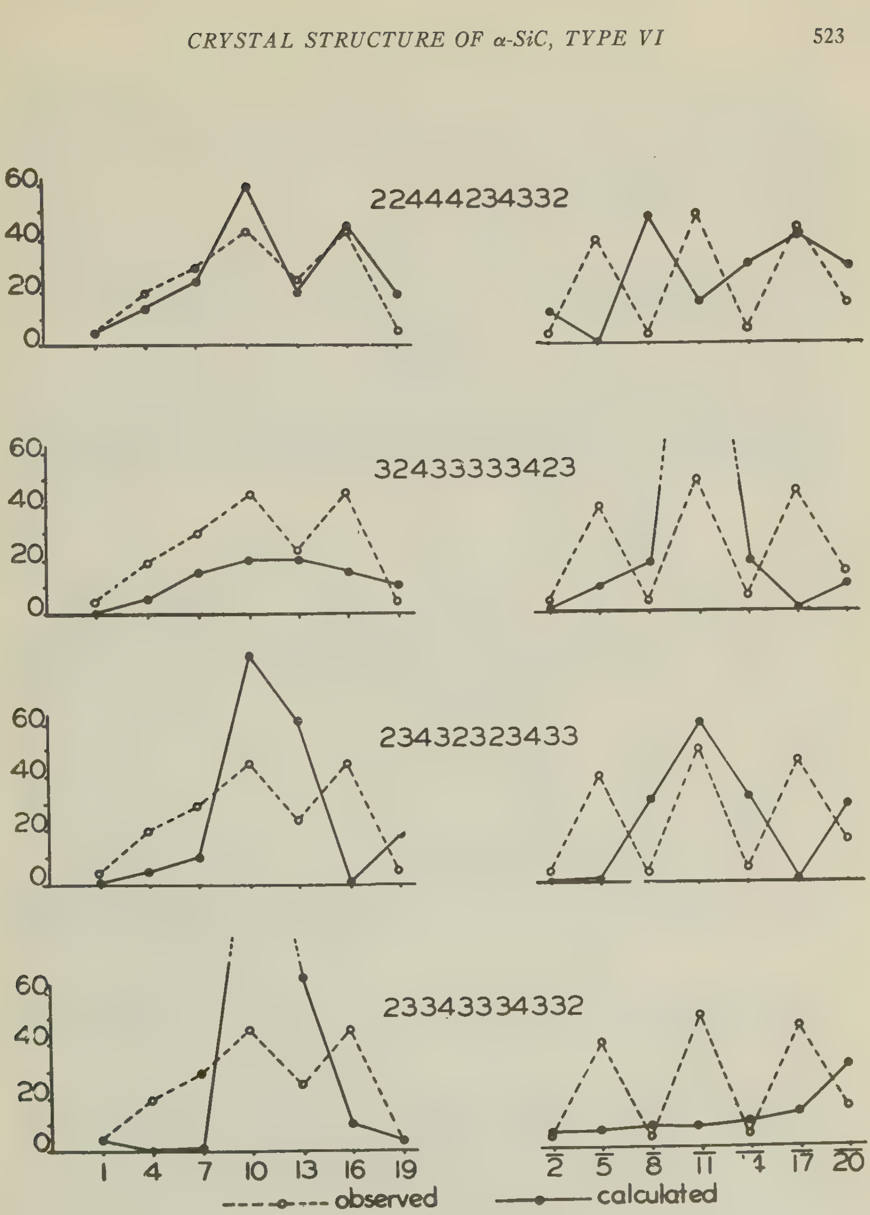

FIG. 2. Observed intensities compared with the calculated intensities for the first 14 reflections of the $10\cdot l$ series of α-SiC, type VI, for the four discarded sequences. Relative intensities are plotted vertically, and values of l are plotted horizontally.

Three main series of reflections appear on the 0-level and 1-level Weissenberg photographs about an *a*-axis, namely, the $10\cdot l$, $20\cdot l$ and $21\cdot l$ series (Fig. 1). For these reflections the intensity calculations are relatively simple, the formula being

$$I\alpha[\Sigma f_0 \text{ Si} \cos 2\pi(lz)+\Sigma f_0 C \cos 2\pi(lz+p)]^2+[\Sigma f_0 \sin 2\pi(lz)+\Sigma f_0 C \sin 2\pi(lz+p)]^2.$$

The results obtained with the use of this formula were multiplied by the Lorenz-polarization factor,

$$\frac{1+\cos^2 2\theta}{\sin 2\theta}.$$

It will be noticed that in the intensity formula the Miller indices *h* and *k* do not occur. The value of *l* is the determining factor, and except for differences in f_0 and the correction factor, the calculated values are alike for a given value of *l*, irrespective of *h* and *k*. Accordingly, there is such similarity in the intensities for the three series $10\cdot l$, $20\cdot l$ and $21\cdot l$ that calculations for only one series are sufficient to confirm or reject a possible structure. For the four discarded sequences, intensities were calculated for the first 14 reflections in the $10\cdot l$ series, while for the accepted sequence intensities were calculated for 34 reflections.

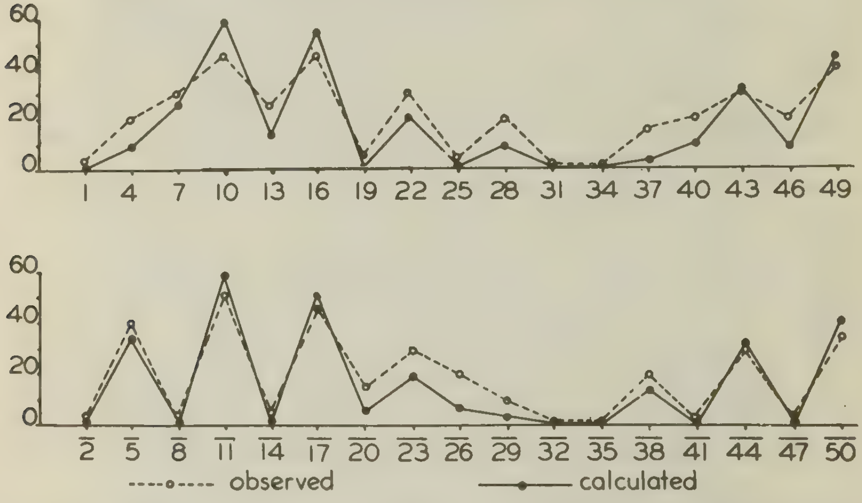

FIG. 3. Observed intensities compared with the calculated intensities for the first 34 reflections of the $10\cdot l$ series of α-SiC, type VI, for the accepted sequence 24243334242.

The results of the calculations are shown graphically in Figs. 2 and 3. Figure 2 shows clearly the marked discrepancies between the observed intensities and those calculated for the four discarded sequences, while Fig. 3 reveals the excellent correlation between the observed values and those calculated for the accepted sequence. The highest calculated value for the accepted sequence was 59,000, which was obtained for the $10\cdot\overline{11}$ reflection. In Fig. 3 this is plotted as 59, and the observed intensity for this same reflection is arbitrarily rated as 50. Two Weissenberg films, the 0-level and 1-level rotations about an *a* axis, were used for the intensity estimates. No corrections for absorption were made. The series $10.l$ and $01.l$, which should have identical intensities, show some minor differences. Accordingly, the intensity values as visually estimated from the films can only be regarded as approximate. However, the intensity variations are great, especially in consecutive reflections such as $10\cdot\overline{2}$, $10\cdot\overline{5}$, $10\cdot\overline{8}$, $10\cdot\overline{11}$, $10\cdot\overline{14}$, etc., where the reflections alternate from very weak to very strong. With such intensity contrasts, even approximate estimates of intensity are sufficient to make a choice between possible structures.

The atomic arrangement of α-SiC, type VI, corresponding to the accepted sequence 24243334242, is derived as follows. On the vertical axis *A* through the origin, 11 silicon atoms are placed on levels separated by the numbers of the sequence, as follows: 0, 2, 6, 8, 12, 15, 18, 21 25, 27, 31, 0. On the vertical axis *B*, through the point 2/3 1/3 0, a similar series begins with the 11th layer, with 11 silicon atoms on 11, 13, 17, 19, 23, 26, 29, 32, 3, 5, 9. On the vertical axis *C*, passing through the point 1/3 2/3 0, a similar series begins with the 22nd layer, with 11 silicon atoms on 22, 24, 28, 30, 1, 4, 7, 10, 14, 16, 20. Above each of the 33 silicon atoms is a carbon atom, at a vertical distance of 1.90Å. This distance represents 1/44 of the length of the *c* axis, while the distance between the silicon layers is, of course, 1/33 of the *c* axis. Accordingly the atomic coordinates for the structure of α-SiC, type VI are as follows:

Hexagonal unit cell; $Z=33$.

11 Si at 000, $0\,0\,2z$, $0\,0\,6z$, $0\,0\,8z$, $0\,0\,12z$, $0\,0\,15z$, $0\,0\,18z$, $0\,0\,21z$, $0\,0\,25z$, $0\,0\,27z$, $0\,0\,31z$.

11 C at $00p$, $0\,0\,2z+p$, $0\,0\,6z+p$, $0\,0\,8z+p$, $0\,0\,12z+p$, $0\,0\,15z+p$, $0\,0\,18z+p$, $0\,0\,21z+p$, $0\,0\,25z+p$, $0\,0\,27z+p$, $0\,0\,31z+p$.

11 Si and 11 C at 2/3 1/3 1/3 plus the above coordinates.

11 Si and 11 C at 1/3 2/3 2/3 plus the above coordinates.

Rhombohedral unit cell; $Z=11$.

11 Si at 000; $2z, 2z, 2z$; $6z, 6z, 6z$; $8z, 8z, 8z$; $12z, 12z, 12z$; $15z, 15z, 15z$; $18z, 18z, 18z$; $21z, 21z, 21z$; $25z, 25z, 25z$; $27z, 27z, 27z$; $31z, 31z, 31z$.

11 C at ppp, $2z+p, 2z+p, 2z+p$; $6z+p, 6z+p, 6z+p$; $8z+p, 8z+p, 8z+p$; $12z+p, 12z+p, 12z+p$; $15z+p, 15z+p, 15z+p$; $18z+p, 18z+p, 18z+p$; $21z+p, 21+p, 21z+p$; $25z+p, 25z+p, 25z+p$; $27z+p, 27z+p, 27z+p$; $31z+p, 31z+p, 31z+p$.

For both hexagonal and rhombohedral unit cells, $z=1/33$, $p=1/44$.

OCTAHEDRAL CRISTOBALITE WITH QUARTZ PARAMORPHS FROM ELLORA CAVES, HYDERABAD STATE, INDIA

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Abstract

The vesicles found in a basalt contain layered quartz inverted from cristobalite with fibers of mordenite projecting from the basalt through and above the quartz. Attached to the free mordenite fibers are well-formed twinned octahedrons of cristobalite, some of which have inverted to quartz. The crystals contain either one or the other mineral. The cristobalite crystals are uniaxial negative, $\epsilon = 1.4862$, $\omega = 1.4841$; $G = 2.382$. The optic axes of cristobalite parallel one of the octahedral diagonals; those of quartz have a scattered orientation. Mordenite fibers formed first in the vesicles. Later, or contemporaneous with mordenite, a layer of massive cristobalite deposited, along with crystals attached to the mordenite fibers. Inversion of the massive cristobalite and some of the crystals followed. The unusual occurrence of cristobalite perched on mordenite indicates that cristobalite formed as an unstable mineral with temperatures not far from 300° or 400°C.

In 1941, B. F. Buie collected specimens of a vesicular basalt from the Deccan volcanics at Ellora caves situated in the northeast corner of Hyderabad State, India. These specimens are characterized by the occurrence in the vesicles of a cotton-like mass of the zeolite mordenite together with well-twinned octahedrons of cristobalite, some of which have inverted to quartz.

The material was studied in the laboratory by Van Valkenburg and the paper was written by him, as Buie is on active duty with the U. S. Army. The work was done under the direction of Professor Esper S. Larsen, and we wish to express our sincere thanks to him and also to Professor Cornelius S. Hurlbut, Jr., and Mr. F. A. Gonyer of Harvard University and to Mr. John C. Rabbitt of the U. S. Geological Survey for many helpful suggestions.

Description

The vesicles in the specimens range in size from 1 to 10 cm. across. A typical one has a wall covered with a layer of quartz about 5 mm. thick. This layer has a waxy luster and shows a pitted contact with the basalt. Fibers of mordenite project from the basalt through and above the quartz giving the quartz surface a cotton-like appearance. Octahedrons of cristobalite, some of which have inverted to quartz, are perched on the mordenite fibers (Fig. 2). The general relations are shown in Fig. 1.

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* Contribution from the Department of Mineralogy and Petrography, Harvard University, No. 269.

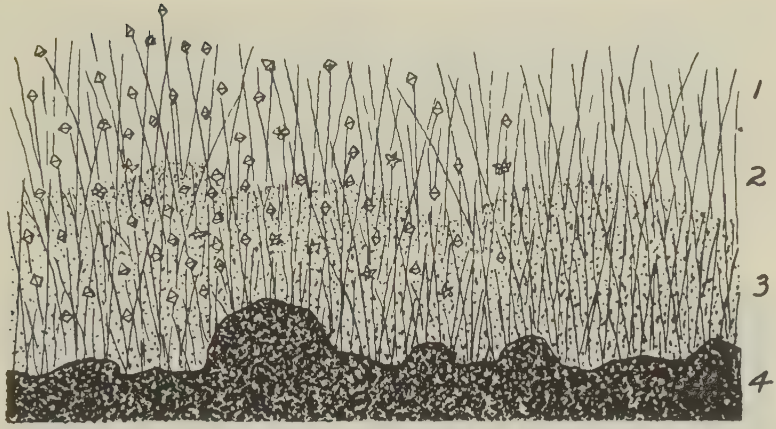

FIG. 1. An idealized cross section of a vesicle wall showing (1) crystals of cristobalite and quartz paramorphs perched on mordenite, (2) zone just above quartz layer where only quartz paramorphs occur, (3) layer of quartz inclosing crystals of quartz paramorphs perched on mordenite, (4) basalt with pitted contact. (Drawn by E. A. Schmitz.)

FIG. 2. Cristobalite and quartz perched on mordenite.

CRISTOBALITE AND INVERTED QUARTZ

Both quartz and cristobalite occur in twinned octahedrons that are identical macroscopically. In a paper following this Dr. Wolfe describes the crystallography of the two minerals. Crystals that occur near the outer mordenite ends are clear and have few inclusions; those near the walls of the quartz are milky and contain abundant inclusions of mordenite. The layered quartz has inverted from cristobalite, as the crystals on the drusy surface are twinned octahedrons. A thin section shows octahedral quartz crystals attached to mordenite but encased in massive inverted quartz. Adjacent to the quartz surface, paramorphs of quartz after cristobalite are perched on the mordenite. In this part all the crystals have inverted and no cristobalite was seen; near the mordenite ends, however, cristobalite and the quartz octahedrons occur together. One crystal may be cristobalite and the next may have inverted. Under the microscope it was seen that every crystal is composed entirely of one mineral or the other. No evidence was found to explain this unusual occurrence. It seems probable that inversion proceeded rapidly after a nucleus of quartz had inoculated the cristobalite. Inversion from cristobalite to quartz involves a volume change of 10 per cent and one might expect fracturing during inversion but careful examination of the quartz paramorphs shows no fractures.

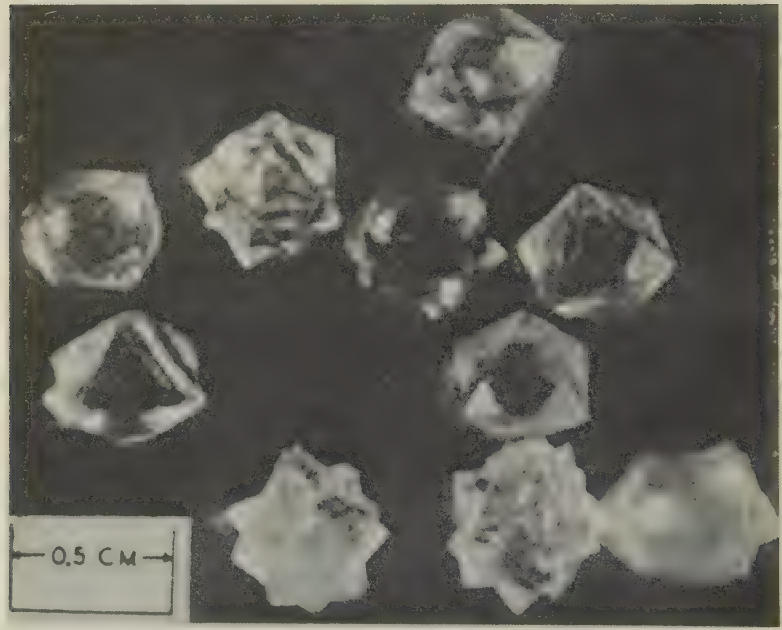

FIG. 3. Cristobalite octahedrons and twins shown in reflected light.

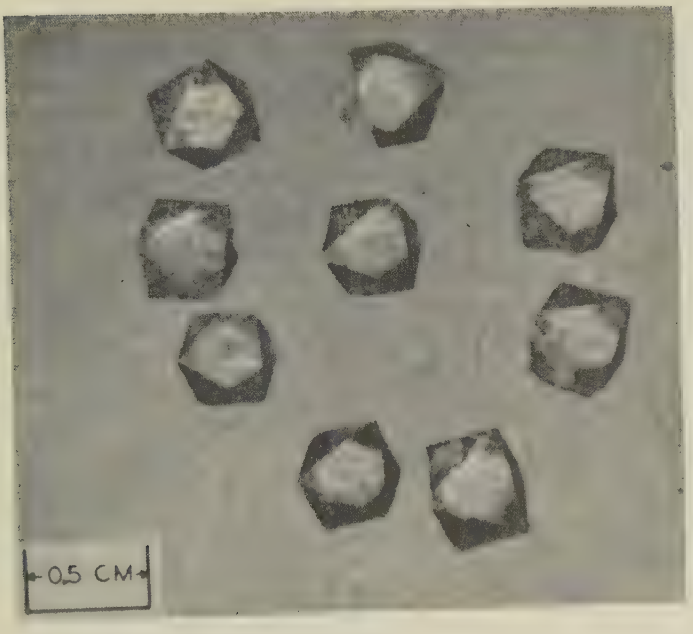

Fig. 4. Cristobalite octahedrons in transmitted light showing etching on faces.

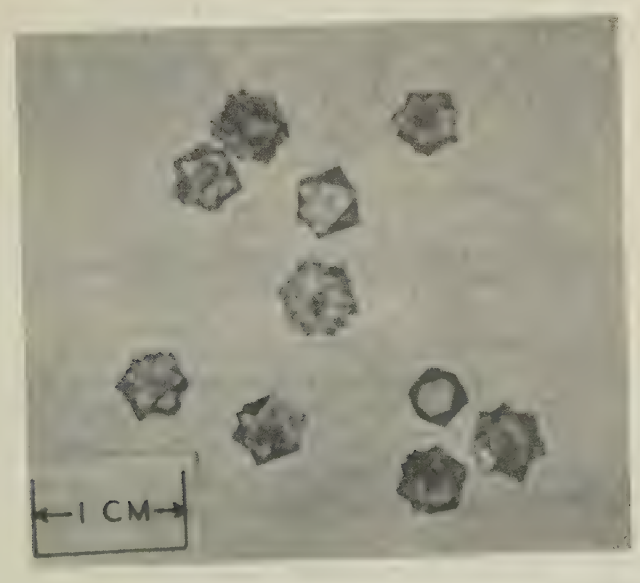

Fig. 5. Quartz and cristobalite octahedrons shown in transmitted light.

The cristobalite octahedrons and twins are about 0.5 mm. in diameter and are transparent to milky white, depending on the amount of mordenite inclusions present (Fig. 3). Etching on the octahedral faces of both quartz and cristobalite is common (Fig. 4). The crystals of cristobalite are optically uniaxial negative with $\epsilon = 1.4862$ and $\omega = 1.4841 \pm 0.001$. The specific gravity as determined in heavy liquids is 2.382 ± 0.003. The optic axis is parallel to one of the diagonals of the octahedron. A thin section was made of 30 untwinned cristobalite crystals with each crystal lying on an octahedral face. In thin section the faces have the outline of a triangle. Using the Fedorov stage for each grain, the vertical plane including the optic axis was turned until its trace on the stage (in 0 position) was normal to the N-S cross hair. For reference position the edge making the smallest angle with the N-S cross hair and with the opposite corner of the triangle to the right was used. The optic axis was located in the usual way. The angle between the optic axis and an octahedral edge was found to average 53°. Figure 6 shows the stereographic plot of the optic axes

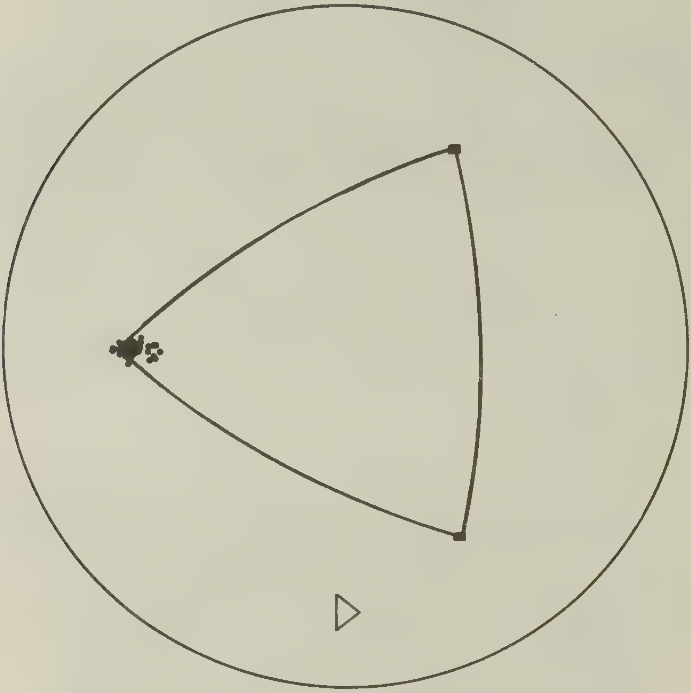

Fig. 6. A stereographic plot of 30 untwinned cristobalite crystals showing concentration of optic axes about an octahedral axis.

with respect to the crystal axes. The squares represent the octahedral axes. One of the octahedral axes was chosen to show the concentration of the optic axes as any one of the octahedral axes could have been used as a reference to show the concentration. The triangle at the bottom of the diagram shows the orientation of the face with respect to the vertical cross hair.

X-ray powder photographs using Cu/Ni(CuKα=1.539 Å) radiation were taken of known critobalite from the Miocene lavas of the San Juan Mountains of Colorado and of the crystals described in this paper. The two photographs are identical and their "*d*" spacings are given below. For comparison the "*d*" spacings of an alpha cristobalite (1) are given.

	"*d*"		*I*	
Line	1	2	1	2
1	4.03	4.04	10	10
2	3.34	3.13		7
3	3.11	2.89	7	8
4	2.81	2.48	8	9
5	2.46	2.11	9	
6	2.10	2.02		
7	2.01	1.93		
8	1.92	1.87		
9	1.86	1.69		
10	1.681	1.61		
11	1.602	1.57		6
12	1.526	1.53		
13	1.483	1.494		
14	1.431	1.430		
15	1.415	1.400		
16	1.394	1.370		
17	1.359	1.339		
18	1.326	1.300		
19	1.293	1.273		
20	1.273	1.235		
21	1.198	1.203		
22	1.092	1.181		
23		1.095		

1. Ellora Caves and San Juan cristobalite.
2. Alpha cristobalite.

"*d*" = spacings in Å units, *I* = relative intensity.

Powder photographs were also taken of the quartz paramorphs and no cristobalite was seen to exist in the crystals. The crystals have the optical properties of quartz but retain the octahedral habit of cristobalite. Undulatory extinction is common in many of the crystals and their twins.

The optic axes of the quartz paramorphs have a scattered orientation with respect to the octahedral axes. Figure 7 shows an equal-area plot of about 100 optic axes measured on the Fedorov stage in the same way as were the cristobalite axes. Figure 8 shows a contoured diagram of Fig. 7 in which the segment has been rotated through two 60° arcs. The numbers represent relative concentrations of the optic axes; the squares are the octahedral axes and the circles the poles of the positive faces. The area with concentration 8 shows significant concentration of axial points, but the point location has no simple relation to the octahedral axes or faces. There is a suggestion of slight concentration in a zone midway between the center and the circumference of the diagram but not enough to say that a preferred orientation exists. On the inversion of alpha to beta cristobalite, an inversion that takes place rapidly with a small adjustment in cell structure, one of the octahedral axes becomes the axis of the

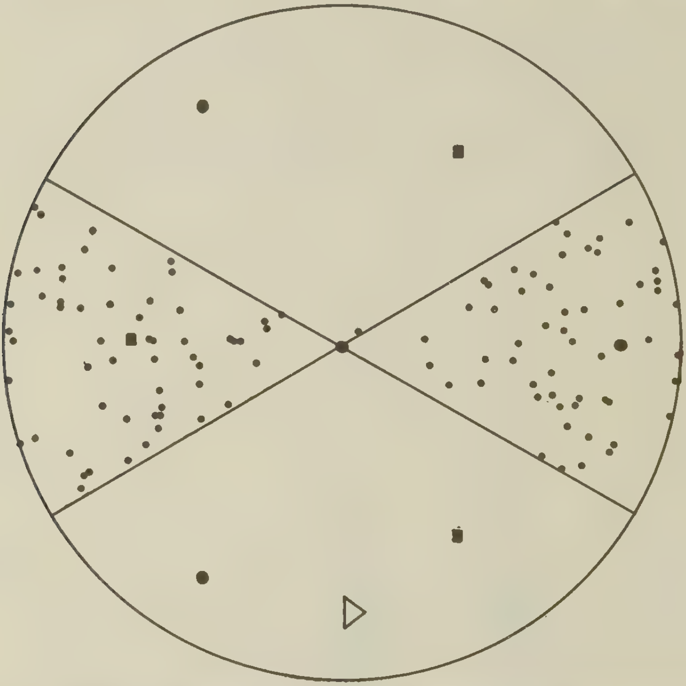

FIG. 7. An equal-area plot of about 100 optic axes of quartz paramorph lying on an octahedral face shows a scattered orientation of the axes. The squares represent octahedral axes and the circles the octahedral faces. The triangle indicates the orientation of the crystals with respect to the vertical cross hair of the microscope. The edge is vertical that gives the smallest extinction angle.

inverted crystal; however, on the inversion of beta cristobalite to quartz, an inversion which is commonly sluggish and takes place with a large volume change, the orientation of the quartz is not closely controlled by the old structure.

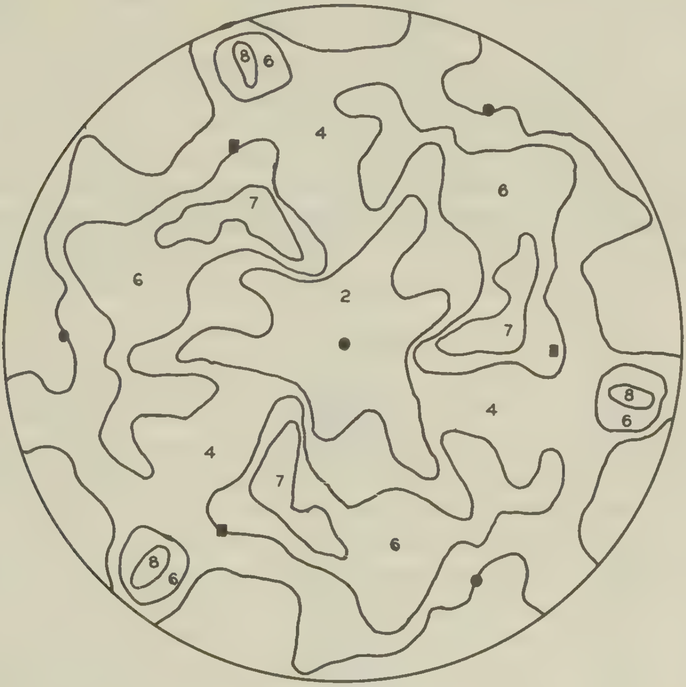

Fig. 8. A contoured diagram of Fig. 7 with the segment rotated through two 60° arcs, showing relative concentrations of optic axes.

Mordenite

Crystals of mordenite completely cover the outer surfaces of the vesicle. The needles of mordenite are white to transparent and have a maximum diameter of about 0.08 mm. and an average diameter of about 0.02 mm. Under the microscope the needles show faint birefringence, parallel extinction, and negative elongation. A thin amorphous film coats the needles, making it difficult to determine the indices. One index was found to be 1.481 ± 0.003. The amorphous film has an index lower than typical mordenite. A formula has been derived from the following chemical analysis by F. A. Gonyer on mordenite fibers which are slightly contaminated by the amorphous film. The unit cell contains 24 oxygen atoms (2).

	%	Mol. number	Oxygen	Bases	Atomic numbers
SiO_2	64.24	1.070	2.140	1.070	9.59
Al_2O_3	14.47	.142	.426	.284	2.52
Fe_2O_3	1.23	.008	.024	.016	0.14
CaO	4.49	.080	.080	.080	0.71
Na_2O	1.55	.027	.026	.052	0.46
H_2O	13.84	.769		1.538	14.01
Total	99.82		2.696		

Formula $= Ca_{0.71}Na_{0.46}Al_2(Al_{0.52}Si_{9.59})O_{24} + 14.01H_2O$.

An *x*-ray powder picture of mordenite, using Cu/Ni_9($CuK\alpha = 1.539$ Å) radiation shows the following "*d*" spacings in Å units, with a few line intensities:

Line	"*d*"	*Relative Intensity*
1	8.25	10
2	6.47	7
3	5.72	6
4	4.48	8
5	3.87	9
6	3.84	
7	3.43	
8	3.35	8
9	3.18	
10	2.85	
11	2.67	
12	2.55	
13	2.50	
14	2.03	
15	1.945	
16	1.86	
17	1.792	
18	1.652	
19	1.515	
20	1.435	

CONDITIONS OF FORMATION

This unique association of cristobalite perched on mordenite fibers indicates that temperatures were not high when the cristobalite formed. The mordenite has a fresh appearance and shows no alteration on contact with quartz and cristobalite. The basalt next to the quartz-mordenite filling is altered for only about two millimeters. Fenner (3) points out that cristobalite can form below its inversion point (1470°C.) and cites several laboratory experiments in support of this view. He says further that "any set of conditions which will bring together quantities of ungrouped SiO_2 molecules in such a manner as to favor their rapid assemblage in definite groupings without giving time for perfect equilibrium to be reached (as in the reaction of vapors); or which will suddenly bring a system in which equilibrium prevails into new conditions, at the same time introducing obstacles to the establishment of a new equilibrium (as in the sudden chilling of a melt), will favor the deposition of unstable forms." Larsen (4) has shown that cristobalite found in gas cavities of the San Juan lavas of Colorado formed much below its inversion point and he believes the lavas were extruded at a temperature below 900°C.

In the material here described the cristobalite was deposited later than, or contemporaneous with, mordenite and both were deposited in the vesicles of a basalt. The cristobalite must have formed at a temperature well below its range of stability (above 1473°C.) and the association with mordenite indicates a temperature not far from 300° or 400°C.

PARAGENESIS

After the basalt solidified, fluids in the vesicles, some of which may have moved upward from the magma, altered the walls and deposited mordenite, followed and probably accompanied by cristobalite. Massive drusy cristobalite formed on the walls and on the mordenite needles after all or most of the mordenite had formed. Later, the massive cristobalite on the walls, those crystals near the walls, and part of the crystals near the outer ends of the mordenite, inverted to quartz. Alteration of the walls has furnished the silica and alumina for the mordenite, but most of the silica for the cristobalite must have been brought in as the amount of silica deposited is too great to have been derived by the small amount of alteration of the walls.

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CRYSTALLOGRAPHY OF CRISTOBALITE FROM ELLORA CAVES, INDIA

C. W. Wolfe,
Boston University, Boston, Massachusetts.[*]

Thirteen simple octahedral crystals of low cristobalite from Ellora Caves were measured, giving readings for 52 separate octahedral faces. The average rho angle for all readings was 54°42′. The closeness of this average reading to the calculated octahedral angle of 54°44′ is surprising since none of the faces gave satisfactory reflections. The rho values ranged from 52°01′ to 56°53′, although most were within one degree of the average. The readings for the phi azimuthal angle varied from 87°11′ to 93°07′. The calculated angle is 90°00′.

Although the crystals appeared perfect, etching was seen under the binocular microscope. Not one of the measured 52 faces gave clear, single signals on the goniometer. Signals for each face varied from two to four in number and were generally indistinct. Orientation of the crystals was consequently difficult. The light was not reflected from each face as a unit but generally from small triangular regions along one of the edges of the face. The lines bordering these reflecting spots were always parallel to the edges of the face. The reflections were studied carefully to determine whether any systematic departure from isometric symmetry could be discovered, but none could be noted. It was impossible to determine whether the poor reflections were the result of etching or of distortion due to inversion, although the latter seems more likely.

Considering the poor quality of the reflections, the angular values discovered for these cristobalites were not sufficiently critical to fix the symmetry or to even suggest that any symmetry other than isometric is probable.

TWINNING

At least 50 per cent of the cristobalites showed penetration twins on the simple octahedral law. The apparent perfection of the twinning was not confirmed very well by the measurements. Four twinned crystals were measured and plotted on a stereographic diagram. Each plot was superimposed over a diagram with the face poles in their calculated positions. Although perfect identity was never realized, the deviations were not great, nor were they systematic in character. That twinning occurred when the crystals were at a higher temperature and were truly isometric seems clear.

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[*] Contribution from the Department of Mineralogy and Petrography, Harvard University, No. 270.

QUARTZ PARAMORPHS AFTER CRISTOBALITE

Seven crystals which had been separated as quartz paramorphs after cristobalite by Van Valkenburg were measured, and similar results to those for low cristobalite were obtained. The only difference that could be noted was a general deterioration of the quality of the signals in the quartz paramorphs. The angular relationships of both the simple and twinned crystals seemed entirely comparable to those for low cristobalite. Instead of isolated areas reflecting imperfect signals as in low cristobalite, each face of the paramorphs gave one large, diffused light reflection with no concentration of light in any part of the signal. This fact seems to indicate that continued inversion from high cristobalite to quartz produced such distortion of the lattice that the formerly perfect faces became highly irregular.

NOTES AND NEWS

An Appeal to American and Canadian Mineralogists

Fellow Mineralogist:

The Mineralogical Museum of the University of Liége, Belgium, was destroyed by fire on September 7, 1944, the day the Germans were leaving the city. On February 20, 1945, the Council of the Mineralogical Society of America voted to sponsor the proposal that a continental committee be established to appeal for minerals to restore the Liége Museum and appointed Charles Palache as chairman to organize the committee. It is not known at this time what damage has been suffered by other mineralogical collections in the allied countries, but the committee was instructed by the Council to seek to learn the facts and it will consider the needs of other institutions as far as may be possible.

The purpose of this letter is to launch a continent-wide drive for *mineral specimens* to be presented to the University of Liége as soon as shipping becomes available (possibly by next fall). The committee is chiefly concerned with gathering specimens and is, therefore, not soliciting funds. Money contributions will, however, be accepted by the Secretary; they will be used to cover expenses or to purchase mineralogical books and equipment.

The Belgian-American Educational Foundation of New York (Dr. P. C. Galpin, President) has kindly agreed to pay for the shipment of the gifts from America to Belgium.

Three kinds of specimens are needed: (1) display; (2) teaching; (3) research. The need may be estimated from an appraisal of the damage suffered by the museum (see Appendix). Reprints of scientific papers had better be mailed directly by the authors to the Museum in Liége when fourth-class mail service is re-established to Belgium. If other gifts are contemplated (such as books, wooden models, apparatus), special arrangements should be made through the Secretary.

How to Prepare Your Specimens: Each specimen should be carefully wrapped with its original label signed by donor. A catalogue filing card (3″×5″), white, preferably unruled and punched, should be typed, giving: name of mineral, locality, size of specimen in centimeters and a short description of features illustrated, name and address of donor. If you do not happen to have such cards, please use pieces of plain white paper of the same size. The filing cards will be assembled into a catalogue to be shipped to Liége along with the collection.

Specimens should be sent prepaid to one of the curators: Mr. Charles R. Toothaker, The Commercial Museum, 34th Street below Spruce, Philadelphia 4, Pa., U.S.A., or Dr. V. Ben Meen, Director, Royal Ontario Museum of Mineralogy, Toronto 5, Canada. The corresponding filing cards should be mailed (separately) to the same address.

In the improbable event that too many specimens of one species are received, the committee will use its discretion and offer such samples to other devastated museums where they will do the most good or, if the donor desires, will return them to him by express, C.O.D.

The continental committee would welcome the formation of local committees under the sponsorship of local mineralogical societes or geological departments of colleges and universities. Two such committees are already functioning, under the sponsorships of the Philadelphia Mineralogical Society and of the Walker Mineralogical Club of Toronto.

In conclusion the undersigned wish to express their confidence that all mineral lovers of America will want to do their utmost to make this project a success.

Charles Palache, *Chairman*, Harvard University, Cambridge 38, Massachusetts.

J. D. H Donnay, *Secretary*, 2926 New Road, Wilmington 165, Delaware.

Hugh Alexander Ford
Edward P. Henderson
Walter F. Hunt
V. Ben Meen
Arthur Montgomery
Joseph Murdoch
Frederick H. Pough
Austin F. Rogers
S. J. Shand
Charles R. Toothaker
R. C. Vance
C. D. Woodhouse

Appendix

Extent of the Damage Done to the Liège Mineral Collections

(1) DISPLAY MATERIAL: (a) Systematic collection: 3000 specimens lost out of 4500. (b) Belgian collection: 545 specimens lost out of 625. (c) Congolese collection: 39 specimens lost out of 395. (d) Cesàro type-material collection: Rhisnes calcites, 90 per cent lost; Vesuvius and Monte Somma minerals, 90 per cent lost; the remainder saved. (e) Meteorite collection: saved. (f) Didactic exhibits: gem collection saved; the other collections either completely or partially lost; the collections of twins and paramorphs greatly damaged; those of alterations and associations lost.

(2) TEACHING MATERIAL: A total loss. This material included 1850 specimens on exhibit, plus study, laboratory, and examination sets. The igneous rocks with thin sections are lost. Wooden models: 30 remain out of about 900. Three goniometers are left out of 13; three microscopes out of 19. All the teaching aids, projector, photographic and microphotographic equipment, furnace, analytical and specific gravity balances, etc., are destroyed.

(3) RESEARCH MATERIAL: For research and comparison, representatives of many species are needed. Only 455 species are still represented. Some of the most expensive research equipment has been saved.

(4) MINERALOGICAL LIBRARY: A number of periodicals have been saved. All the books and the large collection of reprints were lost.

PLATINUM CRUCIBLE SUBSTITUTES UNDER WAR LIMITATIONS

A. R. V. Arellano,
Mexico City, Mexico.

The establishment of a small mineralogical laboratory under war conditions presented innumerable problems, of which the most formidable one appeared to be the acquisition of a platinum crucible for sodium carbonate fusions and silica determinations.

Platinum has an official price of $35 per ounce troy (31.1 grams) and since a suitable crucible need not weigh much over 40 grams, there apparently should not be a prohibitive expense involved. However, platinum is absent from the free market, so that jewelers in Mexico City are glad to pay 35 pesos (about $7) per gram. A 40 gram crucible would thus cost about $300, well beyond the means of a small laboratory.

Accordingly, at the suggestion of Dr. W. F. Foshag, it was determined to make a gold crucible thick enough to permit handling. Fine gold, 99% or better, is sold by the bank of Mexico for industrial uses for about $1 per gram. The crucible, with cover, weighed 65 grams and cost close to 350 pesos. It proved to be very satisfactory for hydrofluoric acid evaporations, as two successive blank runs showed no appreciable loss in weight. After many carbonate fusions and HF evaporations and blastings the loss in weight was only 13 milligrams.

Careless blasting during a carbonate fusion perforated the crucible, which was taken to the goldsmith for repairs. While there a small amount of palladium became available. Tests run on this material[1] indicated that it was pure enough so that the main mass of it was merely scorified with boric acid and potassium nitrate. During these tests it was found that the more accessible texts and hand-books of chemistry and assaying contain very little precise data on the solubility of palladium in sulfuric acid, therefore it may be of interest to note that 80% H_2SO_4 at temperatures up to 160°C. does not appreciably dissolve palladium, either pure or in a silver alloy.

The palladium was then alloyed with four times its weight of gold and from this a 23 ml. palau crucible was prepared. It weighed 47 grams with cover. The crucible lost only about 0.5 mg. per double HF evaporation. Palau crucibles have proven very satisfactory for sodium carbonate fusions.[2] The higher melting point, almost 1400°C., is an advantage over gold and the carbonate cake is more readily freed from palau than from platinum or platinum-iridium. Washington found a 32 g. palau crucible to lose an average of 0.2 mg. per run during 47 carbonate fusions.

The cost of this palau crucible was 350 pesos (palladium @ 9.50 and gold @ 5.50 pesos per gram), which is actually less than a pure gold crucible would have cost, because of the smaller weight necessary to give sufficient rigidity for handling.

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[1] Courtesy of Societé Affinage de Metaux, MM. Antoine Pagés, Director and Paul Tapie, Assayer.

[2] Washington, H. S., Note on crucibles used in rock analysis. *Jour. Wash. Acad. Sci.*, **11**, 9–13 (1921).

HOLLOW PRISM FOR GONIOMETRIC CALIBRATION OF REFRACTIVE INDEX MEDIA

Richard F. Cogswell,
Petrographic Laboratory, AC Spark Plug Division, General Motors Corporation, Flint, Michigan.

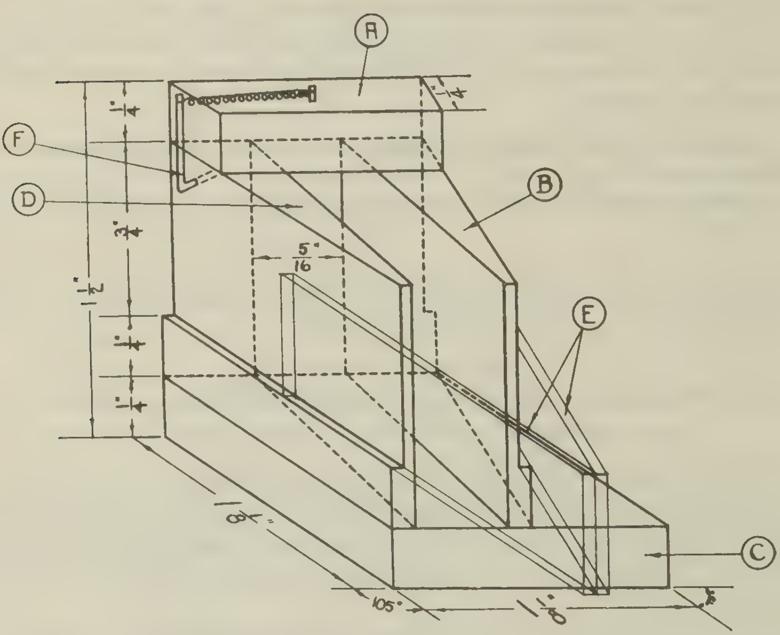
The speed with which refractive indices of media over 1.710 can be determined using an ordinary spectrometer depends largely upon the hollow prism. Several types of prisms have been tried in this laboratory and the one shown in the accompanying sketch has proven the most satisfactory.

The fixture is made of brass, excepting the spring which is of steel; the stock should be of sufficient thickness to insure rigidity. The top brace bar (A), the right prism wall (B), and the base (C) are held together by a $\frac{1}{8}''$ pin of $1\frac{1}{2}''$ length. The left prism wall (D) is similarly pinned to the top brace bar and the base, but in a manner to allow outward swing of the prism wall so that the prism glasses (E) may be readily cleaned. The spring anchor (F) is placed at an angle to the prism wall (D), so that when the wall is swung back the spring passes the maximum extension point and holds the wall open.

A hole through the fixed prism wall (B) near the base, is tapped to accommodate an adjusting screw. An accurate 30° prism angle can be obtained with the minute adjustments afforded by this screw whose end abuts against the inner surface of the pivoted prism wall (D).

A standard 26×45 mm. microscope slide, chosen with parallel faces, is cut lengthwise to form the two faces of the hollow prism, between which faces a drop of the liquid to be measured is suspended. Each of these faces is held securely against its prism wall by a thin metal plate, each plate being anchored to the prism wall by two screws above and two below the glass.

The fixture may be secured to the stage in the proper working position



Sketch of Hollow Prism and Fixture

Fig. 1

by means of two aligning dowel pins built into the base, which fit into appropriate holes provided in the goniometer table.

The rigidity of the fixture may be determined by opening and closing the prism faces and measuring the included angle several times. For example: It has been found that values vary from 30° 3′ to 30° 3.5′. Thus, for rough calibration, the angle need be measured only once. Even after removing sulfur and various evaporation residues of high index media, the change in the prism angle is very slight upon re-closing the faces.

The Mineralogy of the Tin Mines of Cerro de Llallagua, Bolivia, by Samuel G. Gordon in the *Proceedings of the Academy of Natural Sciences of Philadelphia*, vol. **96**, 270–359 (1944), gives an interesting and comprehensive report on the minerals of this important region which has produced more than 350,000 tons of tin. The paper treats of the general geology of the region, sequence of mineralization, history of the deposit, and a description of the minerals with emphasis placed on the crystallographic forms observed. Many new forms were found. The minerals occur in a network of veins in a quartz porphyry less than a mile wide. While about 60 minerals are described special attention was given to the less common species yielding new mineralogical data. A list of the minerals described in considerable detail includes the following: stannite, greenockite, pyrrhotite-troilite, bismuthinite, marcasite, boulangerite, andorite, rutile, monazite, apatite, crandallite, wavellite, the vauxite group, and hisingerite.

In recent months the Mineralogical Society of America has lost through death one correspondent (Alexander E. Fersman, May 20, at the age of sixty-two years) and two fellows (Edward Wigglesworth, May 6, age fifty-nine, and Florence Bascom, June 19, at the age of eighty-two years). Memorials will be published in the Journal at a later date.

Word was received some time ago of the liberation of Professor Frantisek Slavik, of Charles University, Prague, Czechoslovakia, after two years spent in German prisons and concentration camps. He is in good health and sends his hearty greetings to his numerous American colleagues.

Dr. Alexander N. Winchell, professor emeritus of Geology at the University of Wisconsin, has accepted a position as resident consultant to the American Cyanamid Company at Stamford, Connecticut.

Dr. Waldemar T. Schaller of the United States Geological Survey, past president of the Mineralogical Society of America, and recipient of the Roebling medal in 1938 has been elected Honorary Member of the Mineralogical Society of Great Britain.

BOOK REVIEWS

HANDBOOK OF MINERAL DRESSING, ORES AND INDUSTRIAL MINERALS by ARTHUR F. TAGGART. John Wiley and Sons, Inc., New York (**1945**) $15.00. Price to students $12.00.

This publication represents a completely revised and greatly expanded edition of the *Handbook of Ore Dressing* which was issued in 1927. It is stated in the preface that this is Volume I. Volume II which will follow will treat of the preparation of fuels and the methods by which metalliferous and nonmetallic concentrates are rendered into primary-consumer products. In other words, ore dressing is being expanded to include the entire field of primary mineral technology. This will prove to be an outstanding milestone in the historical development of mineral technology. The willingness of the metallurgist to abandon the treatment of ores and confine his attention entirely to metals and their alloys has been increasing steadily during the past decade. The metallurgist is rapidly becoming exactly what the term denotes—a worker in metals. The field of primary ore treatment tends to become more and more isolated and may soon be recognized as a definite division of mineral technology in which ore dressing, furnace operation, coal preparation, and petroleum refining become component parts of equal importance. This trend has been in evidence for years but Professor Taggart appears to be the first to appreciate its ultimate effects.

The book represents a major advance in size and content paralleling the growth in the importance of the arts of mineral preparation since 1927. The format has been changed and in place of continuous pagination the volume is divided into 22 sections, each one of which is paged separately. Pages total over 1900 and the size has been increased. Flexible binding, excellent paper and typography make the volume an outstanding example of bookmaking, particularly for these times. Thirteen contributors, recognized leaders in their fields, have prepared certain sections, specially those dealing with processes. This adds much to the authenticity and value of the volume.

The mineralogist who so frequently uses the physical properties of minerals as means of identification will be surprised at the extraordinary skill and elaborate equipment with which the engineer uses these same properties to separate the valuable mineral from its worthless associates. Specific gravity is by far the most important, but electrical conductivity, magnetic attractability, and the complex physical-chemical relationships involved in surface wetting and amalgamation are also utilized.

The text contains a complete description of practically all those processes and types of equipment employed in the diverse operations of mineral preparation. Processes range from hand sorting to the most recent developments in flotation. The value of practical data is constantly reinforced by a sound discussion of theory. Performance data and the reproduction of flow sheets from many operations add to the practical utility of the volume. It is difficult to isolate outstanding contributions in the wealth of data presented but to the reviewer the discussion of the principles of flotation appears to be a model of concise and accurate technical writing. As long as the production of minerals is motivated by profit, economic factors will control technical operations. The emphasis which is placed on the economics of production and processing accents this important consideration. Comparatively little data are supplied concerning the properties of minerals and part of that available is not entirely free from error. To the mineralogist this will appear to be a deficiency which is particularly striking in view of the detailed and complete review which is supplied for the principles of mathematics.

The decline in the reserves of first class ore is constantly forcing industry to turn to lower grade materials, which can be used only after concentration. This trend is of growing importance in every mineral raw material and will be increased in the post-war years due to the depletion of high grade stocks which the conflict has caused. Therefore the value of the service which this handbook can render will constantly increase. The engineer, whether located in the city or in those distant and remote areas where minerals have a way of occurring, will find this encyclopedic volume an invaluable source of data concerning the construction and operation of equipment. The research worker will find it to be a mine of information upon which future progress may be dependent.

W. M. Myers

DICTIONARY OF GEMS AND GEMOLOGY by Robert M. Shipley, assisted by Anna M. Beckeley, Edward Wigglesworth, and Robert M. Shipley, Jr. Octavo, XII+254 pages. Gemological Institute of America, Los Angeles and Boston (**1945**). Price $5.50.

During the last two decades dealers in gems and gem materials, as well as the general public, have endeavored to become better informed concerning this important and very fascinating branch of mineralogy. Through the activities of the Gemological Institute of America and the Gemological Society many dealers have been able to secure systematic and authoritative instruction by correspondence, through regional discussion groups, and by actual laboratory experience. That the general public is also eager to become better informed is evidenced by the organization of many local mineralogical clubs, by the development of amateur lapidaries, and by the increased demand for authoritative texts and articles dealing with gem stones. In these activities and in the discussion of the properties, occurrence, and uses of gem stones many terms, synonyms, and varietal names must of necessity be used which are unfamiliar to members of these groups. To make this information available in compact and convenient form this dictionary has been compiled.

As a forerunner to the dictionary a gemological glossary was published serially, 1934 to 1940, in Gems and Gemology which included approximately 1,700 terms. In the present volume many items have been added so that about 4,000 titles are given, which include definitions, abbreviations, synonyms and varietal names, translations of foreign terms, and short biographical sketches. The book should prove very helpful not only to dealers in and lovers of gem stones but also to professional mineralogists.

Edward H. Kraus

THE OPTICAL PROPERTIES OF ORGANIC COMPOUNDS by A. N. Winchell. Photoprinted. pp. 342+X, figs. 157, 17×25 cm., cloth. Published by University of Wisconsin Press, 811 State Street, Madison, Wisconsin (**1943**), Price $5.00.

Professor Winchell has been gathering optical data on crystalline substances for many years and has published books containing such information for minerals and for inorganic chemical compounds. The book now under review completes the series and furnishes the available data for organic substances and for some naturally occurring organic materials.

Twenty-five years have elapsed since the last volume (V) of Paul von Groth's monumental work "Chemische Krystallographie" was published. This standard reference contained both morphological and optical data on minerals, both natural and artificial, and on inorganic and organic chemical compounds. As W. L. Bragg[1] has so aptly written:

> "This record of the form, preparation, and physical properties of crystals has been the familiar book of reference to all workers on crystal structure, and its influence in the development of the new methods (*in crystallography*) has been immense."

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[1] Bragg, Sir W. H., and Bragg, W. L., *The Crystalline State*, Vol. **1**, p. 271 (1934). The MacMillan Co., New York.

Since 1919 there has been a great increase in the use of the so-called "immersion methods" for the measurements of the indices of refraction of crystalline compounds. As a result of this practice, the recent chemical literature contains very many widely scattered data on organic compounds. It has been necessary for research workers on organic compounds to spend much time in searching for optical data in the abstracts of the American, and the various foreign chemical publications. The present work by Winchell will greatly reduce this effort and will fill a long existing need. The compiler has wisely adopted the system of Beilstein's "Handbuch der organischen Chemie" (4th edition) for arranging the organic compounds. This arrangement should help to introduce optical data and optical-crystallographic methods to a wider audience among the organic chemists. The main divisions used by Winchell are: I—Acyclic Compounds, II—Isocyclic Compounds, III—Heterocyclic Compounds, IV—Natural Products, not assigned places in the three preceding groups. There are certain minor deviations from Beilstein which are explained by the author in the introduction.

The French notation for the principal indices of refraction, used by Winchell, may detract somewhat from the convenience of the book; a notation not commonly employed in English-speaking countries. The method of reproduction and the quality of the paper used serve greatly to increase the difficulty in identifying the subscripts. In treating the data of Bolland and others, the compiler might have made some use of the scheme of Buttgenbach, and others, in presenting the optical data for cleavage flakes.

The rest of the book contains a collection of references to the original literature, and a set of determinative tables. These tables (71 pages), are based on the indices of refraction. They should prove very useful. Following this compilation, there is a chart for the determination of compounds in which the individual substances are arranged according to their index of refraction and birefringence. The text accompanying this chart is rather brief and contains generalizations to which numerous exceptions could be cited and hence are not rigorously correct.

Thus on page 341 he writes: "Then select another grain or fragment that shows the highest interference color (for the given thickness); it will have two indices of refraction, N_g and N_p." Particularly for organic materials which commonly have a high to very high birefringence this is not a very reliable method for selecting a section which will give N_g and N_p. Only by securing a grain of known orientation may reliable results be obtained.

The reviewer recommends this book to all those investigators engaged in the study of organic compounds. It will certainly prove a time saver. The author should be complimented for his energy and interest in assembling and organizing the widely scattered data. It is to be hoped that this book will stimulate a wider interest amongst chemists in the useful methods of microscopic-petrographic research so that in the future a book may be writen containing many more critical data on organic compounds.

George T. Faust

U. S. Geological Survey
Section of Petrology
(Published by permission of the Director, U. S. Geological Survey.)

PROCEEDINGS OF SOCIETIES

ABSTRACTS OF MINUTES OF THE NEW YORK MINERALOGICAL CLUB, INC.

Meeting of December 20, 1944

President James A. Taylor presided with Mr. O. Ivan Lee acting as secretary *pro tem.* Mr. Richard Gaines, a member on furlough from service in Africa and Asia, gave an account of his collecting activities abroad, including a three weeks' furlough trip on foot to the Kashmir sapphire mines.

Mr. Samuel Gordon of the Philadelphia Academy of Sciences spoke on "The Tin Mines of Bolivia" which he has visited three times and illustrated his lecture with colored slides. Mr. Gordon described rare specimens from the various mines and discussed the paragenesis of the ores.

Meeting of January 17, 1945

Mr. Arnold Hoffman spoke on "the Mining of Optical Calcite in California and Montana." These two localities were selected as the most promising by the late Dr. Berman. The calcite at Palm Wash, San Diego County, California, occurs as basal plates, the form ultimately desired for the contemplated application. The speaker emphasized the difficulties of initiating mining operations in desert country without roads.

Meeting of February 21, 1945

Mr. W. F. Dingley of the United States Potash Co. discussed the mining of potash salts in New Mexico. The salts occur in Permian beds a thousand feet or more below the surface. Part of Mr. Dingley's talk was accompanied by a motion picture of the mining and processing of the potash salts.

Meeting of March 21, 1945

The speaker of the evening was Mr. Marcel Pochon who has been engaged in radium research since his work with the Curies during the first decade of the twentieth century. Speaking vividly from first hand experience, Mr. Pochon related the story of the development of the radium industry from its dramatic beginning to the present time with special emphasis on the growth of the Great Bear Lake region in which he has played an essential part.

Meeting of April 18, 1945

The following officers were elected for the ensuing year:

President: Mr. O. Ivan Lee
First Vice-President: Dr. F. H. Pough
Second Vice-President: Mr. Ralph J. Holmes
Secretary: Mrs. Edward J. Marcin
Treasurer: Dr. C. H. Kindle
Directors: Mr. G. S. Stanton and Mr. J. A. Taylor

Mr. Edmund Wise of the International Nickel Co. spoke about "Platinum," its mining, metallurgy, and uses, emphasizing especially the deposits at Sudbury. Among the specimens exhibited were a platinum nugget about two inches in diameter and a sperrylite crystal nearly half an inch in diameter.

Elizabeth Armstrong, *Secretary*

THE PHILADELPHIA MINERALOGICAL SOCIETY

The Academy of Natural Sciences of Philadelphia, January 4, 1945

Dr. W. Hersey Thomas presided, with 54 persons present. The appointment of a committee to aid in the restoration of the mineral collections of the Liége museum was announced, which included Harold Arndt, Charles R. Toothaker, and Dr. J. D. H. Donnay.

Dr. Donnay addressed the society on "Twinning," with lantern slide illustrations. He emphasized the geometrical laws and the reticular control of twinning in terms of the theory of the French crystallographers Haüy, Bravais, Mallard, and G. Friedel. The scope of the talk was limited to the cases of triperiodic twinning (by far the most common) twinning by merohedry, pseudo-merohedry, reticular merohedry, and by reticular pseudo-merohedry.

February 1, 1945

Dr. Thomas was in the chair, with 47 persons present. Dr. John Putnam Marble addressed the society on "Radioactive Minerals as Geological Clocks" with lantern slide illustrations.

March 1, 1945

Dr. Thomas presided, with 46 members present. Dr. Herman Yagoda of the National Institute of Health (Bethesda, Md.) spoke on "Printing Methods for the Analysis of Minerals" which was illustrated by numerous lantern slides in color.

April 5, 1945

Dr. Thomas in the chair, with 47 persons present. Mr. John Cochrane addressed the society on "Industrial Applications of the Lesser Known Elements: Indium, Gallium, Germanium, Thallium, Vanadium, Uranium, Lithium, Rubidium, Cesium, Cerium, Thorium, and Neodymium."

J. S. FRANKENFIELD, *Secretary*

NEW MINERAL NAMES

Viseite

J. MÉLON: Viseite, a new mineral species. *Ann. soc. geol. Belg., Bull.* **66,** 53–56 (1943); through *Chem. Zentr.* (**1943**), II, 995; through *Chem. Abstracts*, **38,** 6244 (1944). "In the examination of the minerals that occur with delvauxite at Visé, a new mineral was found to which the name viseite was given. It occurs in little papillary translucent masses which appear white, bluish-white or yellowish-white. It melts at once with efflorescence in the blowpipe giving a white pearl and coloring the flame green. G. = 2.2, H. = 3–4. Under the microscope the mineral appears transparent, homogeneous and similar to glass. Cleavage is not to be observed. It is isotropic and has $n = 1.530$. Chemical analysis leads to the formula $5CaO \cdot 5Al_2O_3 \cdot 3P_2O_5 \cdot 3SiO_2 \cdot nH_2O$ with n between 25 and 30. The mineral loses its water completely at red heat; the H_2O is not bound as water of constitution."

DISCUSSION. Further work is obviously needed.

MICHAEL FLEISCHER

Jusite

ILSE GRAMLING-MENDE AND GUSTAV LEOPOLD: "Jusite," ein vermutlich neues Mineral! *Neues Jahrb. Min. Monatshefte*, Abt. A (**1943**), 178–184; through *Mineralogical Abstracts*, **9,** 37 (1944).

NAME. For the locality, Jus in the Schwabian Alb, Württemberg.

CHEMICAL PROPERTIES. The mean of two analyses was: SiO_2 40.38, Al_2O_3 8.23, CaO 35.12, Na_2O 1.14, K_2O 2.47, H_2O+CO_2 12.80; total 100.14%. The formula is given as "$(Ca,Na,K)_5(Si,Al)_6 \cdot 5H_2O$." [Should be $(Ca,Na,K)_5(Si,Al)_6O_{16} \cdot 5H_2O$. M.F.]. 8.31% H_2O is lost up to 230° and the remainder more slowly up to red heat.

PHYSICAL AND OPTICAL PROPERTIES. White, radially fibrous. The fine needles give parallel extinction with positive elongation and are probably orthorhombic; n ($\perp$ fiber) 1.558, birefringence low. G. = 2.32. X-ray rotation photographs give a spacing along the needle axis of 3.69Å. The mineral differs from pectolite, thaumasite, ptilolite, inesite, riversideite, radiophyllite, yuksporite and the zeolites.

OCCURRENCE. As a filling of amygdaloidal cavities in a dike of melilitite.

M. F.

Calogerasite (= Simpsonite)

CAIO PANDIÁ GUIMARAENS: Calogerasita, um novo Mineral da Familia dos Tantalatos. *Mineração e Metalurgia* (Rio de Janeiro), **8**, No. 44, 135–136 (1944).

NAME: For João Pandiá Calogeras (1870–1935), Brazilian geologist.

CRYSTALLOGRAPHY: Hexagonal, habit prismatic. Forms observed (0001), (1010) and (1120).

PHYSICAL PROPERTIES: Colorless. G. = 7.10 to 7.30. H. = 6½ to 7. Fracture irregular; cleavage basal, good. Optically uniaxial, negative, with n approximately 2 and birefringence 0.042.

CHEMICAL PROPERTIES: Two samples were analyzed in duplicate by Willer Florencio, giving: #1, Ta_2O_5 73.60, 73.30; Al_2O_3 26.10, 26.20; #2, Ta_2O_5 73.80, Al_2O_3 26.20, 26.10. No columbium or other metals were detected spectrographically. This corresponds to $3Al_2O_3 \cdot 2Ta_2O_5$ or $Al_6Ta_4O_{19}$. The mineral fluoresces golden-yellow in ultraviolet light.

OCCURRENCE: Found in a pegmatite in the municipio Equador, Rio Grande do Norte and Paraiba provinces, Brazil.

DISCUSSION: Calogerasite is undoubtedly identical with simpsonite from Tabba Tabba, Australia, described by Bowley (see *Am. Mineral.*, **25**, 313 (1940); *Dana's System*, Seventh Edition, Vol. I, p. 771). X-ray powder photographs made in the chemical laboratory of the U. S. Geological Survey show that the Tabba Tabba material is identical with samples from Rio Grande do Norte province, Brazil. Bowley's statement that simpsonite is optically positive is incorrect. The name simpsonite has priority; the name calogerasite should be dropped.

M. F.

Genthelvite

J. J. GLASS, R. H. JAHNS, and R. E. STEVENS. *Am. Mineral.*, **29**, 163–191 (1944).

Hydrotungstite

P. F. KERR AND FORD YOUNG. *Am. Mineral.*, **29**, 192–210 (1944).

Mackayite

CLIFFORD FRONDEL AND F. H. POUGH. *Am. Mineral.*, **29**, 211–225 (1944).

Blakeite

CLIFFORD FRONDEL AND F. H. POUGH. *Am. Mineral.*, **29**, 211–225 (1944).

Minnesotaite

J. W. GRUNER. *Am. Mineral.*, **29**, 363–372 (1944).

M. F.

DISCREDITED MINERALS

Durdenite (= Emmonsite)

Clifford Frondel and F. H. Pough. *Am. Mineral.*, **29**, 211–225 (194).

Barthite = Austinite

E. Fischer: Ueber die Kristallform, chemische Zusammensetzung und systematische Stellung des Barthite. *Zeits. Krist.*, **105**, Heft 4, 268–272 (1944).

Re-examination of the type material shows it to be orthorhombic, bisphenoidal with $a:b:c = 0.648:1:0.832$. The original analysis was faulty; the As_2O_5 content reported (64.0–65.7%) must have been the sum of As_2O_5 and CaO, as a new microchemical analysis gave As_2O_5 43.2%, CaO 22.5%. The colorless inner core of the mineral was copper-free, the outer green border contained appreciable copper.

Discussion: The name barthite (1914) has priority over austinite (1935). However, the description of barthite, especially the chemical analysis, was so faulty that priority may be set aside and the name barthite (= cuprian austinite) should be dropped.

M. F.

NEW DATA

Vauxite

Samuel G. Gordon: The mineralogy of the tin mines of Cerro de Llallagua, Bolivia. *Proc. Acad. Nat. Sci. Phila.*, **96**, 279–359 (1944); see *Am. Mineral.*, **7**, 108 (1922). New crystallographic data and three analyses are given. The formula is $FeO \cdot Al_2O_3 \cdot P_2O_5 \cdot 6H_2O \pm 2H_2O$. G. = 2.389.

Paravauxite

Samuel G. Gordon, *op. cit.;* see *Am. Mineral.*, **7**, 108 (1922). New crystallographic data and four analyses are given. The formula is $FeO \cdot Al_2O_3 \cdot P_2O_5 \cdot 5H_2O \pm 5H_2O$. G. = 2.358.

Metavauxite

Samuel G. Gordon, *op. cit.;* see *Am. Mineral.*, **12**, 264 (1927). New crystallographic data and two analyses are given. The formula is $FeO \cdot Al_2O_3 \cdot P_2O_5 \cdot 4H_2O \pm 5H_2O$. G. = 2.345.

M. F.